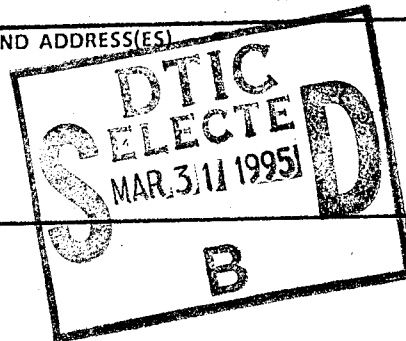


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FINAL
VOLUME 5
BENCH-SCALE LABORATORY INCINERATION
OF SECTION 36 WASTES
(EXPANSION PROGRAM)
ROCKY MOUNTAIN ARSENAL

SEPTEMBER 1988
TASK NO. 17
CONTRACT NO. DAAK11-84-D-0017

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EBASCO SERVICES INCORPORATED

R. L. Stollar and Associates
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1.0 INTRODUCTION

This laboratory investigation is a continuation of research presented in the previous report titled "Bench-Scale Laboratory Incineration of Basin F Wastes at Rocky Mountain Arsenal," (Basin F Laboratory Report) by Ebasco Services Incorporated (Ebasco 1988). The previous research focused on determining the incinerability of Basin F wastes. This report applies the previous research investigation to Section 36 wastes, which include Basin A materials. A summary of the background, laboratory objectives, technical approach, and organization of this report is presented below.

1.1 BACKGROUND

In accordance with the National Contingency Plan, 50 CFR 47912 (1985), the Program Manager's Office (PMO) for Rocky Mountain Arsenal (RMA) Contamination Cleanup is gathering information on the technical and economic aspects of incineration/thermal treatment of contaminated media, particularly soils, as the initial step towards developing a broad remedial action alternative for RMA. To this end, PMO has awarded Task 17 to Ebasco to investigate the incinerability characteristics of Basin F wastes.

PMO chose Basin F material for Task 17 since the soil, sludges, and liquids are considered good candidates for incineration based on available information. In addition, contamination assessment reports for other RMA sites with the potential for treatment by incineration were not available at the initiation of Task 17.

Under the existing Task 17 Scope of Work (Ebasco 1987), Ebasco has investigated the incinerability characteristics of Basin F wastes using a laboratory bench-scale thermal destruction unit. The test data indicated that organics associated with the Basin F wastes (overburden) can be destroyed to better than the 99.99 percent level. The test burns were limited to the overburden samples taken from previously known locations where concentrations of contaminants were found to be the highest. No soil

samples underneath the liner were considered for the laboratory program because past studies have indicated lower concentrations of contaminants in the soils underneath the liner.

Encouraged by the initial test results, PMO asked Ebasco to expand the scope of the existing Task 17 laboratory program to test other areas of RMA. This report, "Bench-Scale Laboratory Incineration of Section 36 Wastes" (Expansion Program), is the result of the expanded scope.

Of all the contaminated sites at RMA, Section 36, perhaps, represents the next most contaminated source after Basin F. Therefore, it was decided to perform incinerability tests on samples from selected areas within Section 36, including Basin A.

1.2 LABORATORY PROGRAM OBJECTIVES

The main objectives of the Section 36 laboratory investigation were fourfold, as summarized below:

- o Determine if the optimum conditions used in the bench-scale incinerator for Basin F samples could also be used to destroy the organic contaminants in Section 36 samples.
- o Investigate primary and secondary reactor conditions to determine their effect on achieving the desired destruction and removal efficiency (DRE) of the selected principal organic hazardous constituents (POHCs), while minimizing the formation of products of incomplete combustion (PICs).
- o Investigate primary reactor residence time and temperatures, with particular emphasis on low temperature volatilization.
- o Identify the partitioning of metals between the bottom residue and the flue gas emissions to the atmosphere.

1.3 TECHNICAL APPROACH

The technical approach involved using equipment that is capable of simulating primary reactor and secondary reactor (afterburner) operation. The equipment used for testing Basin F materials under the existing Task 17 program was used for the Expansion Program. In addition, a fully programmable ashing furnace was used for simulation of primary reactor conditions.

The incineration regime found successful with Basin F contaminated overburden (described in the Basin F Laboratory Report) was used for thermal treatment of contaminated soils from Section 36, specifically Basin A. Basin A soil was used because it has the highest concentration of organic contaminants.

Each feed sample to the bench-scale incineration unit was analyzed for all target compounds reported above the analytical detection limit in the Phase I investigation program conducted by Environmental Science and Engineering (ESE 1987). Similarly, the residue sample from each test burn was analyzed for all target compounds identified in the feed sample. Off-gas samples from each test burn were analyzed for target organics present in the feed sample and for products of incomplete combustion.

Other testing conducted on Section 36 samples included the EP Toxicity test and the Toxicity Characteristic Leachate Procedure (TCLP). Low temperature organic volatilization, metal partitioning, and temperature profiles of the bench-scale unit were also conducted.

1.4 ORGANIZATION OF REPORT

This report begins with a characterization of the Section 36 soils (Chapter 2.0). This is followed by a description of the incineration systems and the test procedures employed (Chapter 3.0). Test results and a discussion are presented in Chapter 4.0. Summary and conclusions are presented in Chapter 5.0.

2.0 BACKGROUND OF SECTION 36 CONTAMINANT SOURCES

This chapter describes the location, topography, and concentration of contaminants of the most heavily contaminated areas in Section 36. Also presented is the sampling procedure used to obtain soil samples for experimentation, as well as a brief description of laboratory experiments conducted on Section 36 soils. In the preparation of this chapter, the project team reviewed a number of documents detailing the history and characteristics of wastes associated with Section 36. References are presented in Appendix E.

2.1 GENERAL SITE DESCRIPTION

There are three major sites of contamination in Section 36: Sources 36-1, 36-3, and 36-17. The location, prior use, topography, and soil characteristics of these sites are described briefly below.

2.1.1 Source 36-1

Basin A, Source 36-1, is located in Section 36, as shown in Figure 2.1-1, and was the original disposal basin for all South Plant Area activities. Until 1956, Basin A was the primary receptor of aqueous waste, with overflow going to Basins C, D, and E. Waste streams from the South Plant Area to Basin A were first passed through lime settling pits to raise the pH and precipitate arsenic and heavy metals. Basin A has covered an area of up to 125 acres at maximum water level. Additionally, during the initial use of Basin F, the majority of waste first passed through Basin A.

Basin A is a topographic low, sloping to the northwest, with inflow coming from the south via channels, pipe flow, and runoff from topographic highs. The basin is underlain by up to 40 ft of alluvium consisting of inter-bedded sands, silts, and clays. The alluvium is generally saturated and the

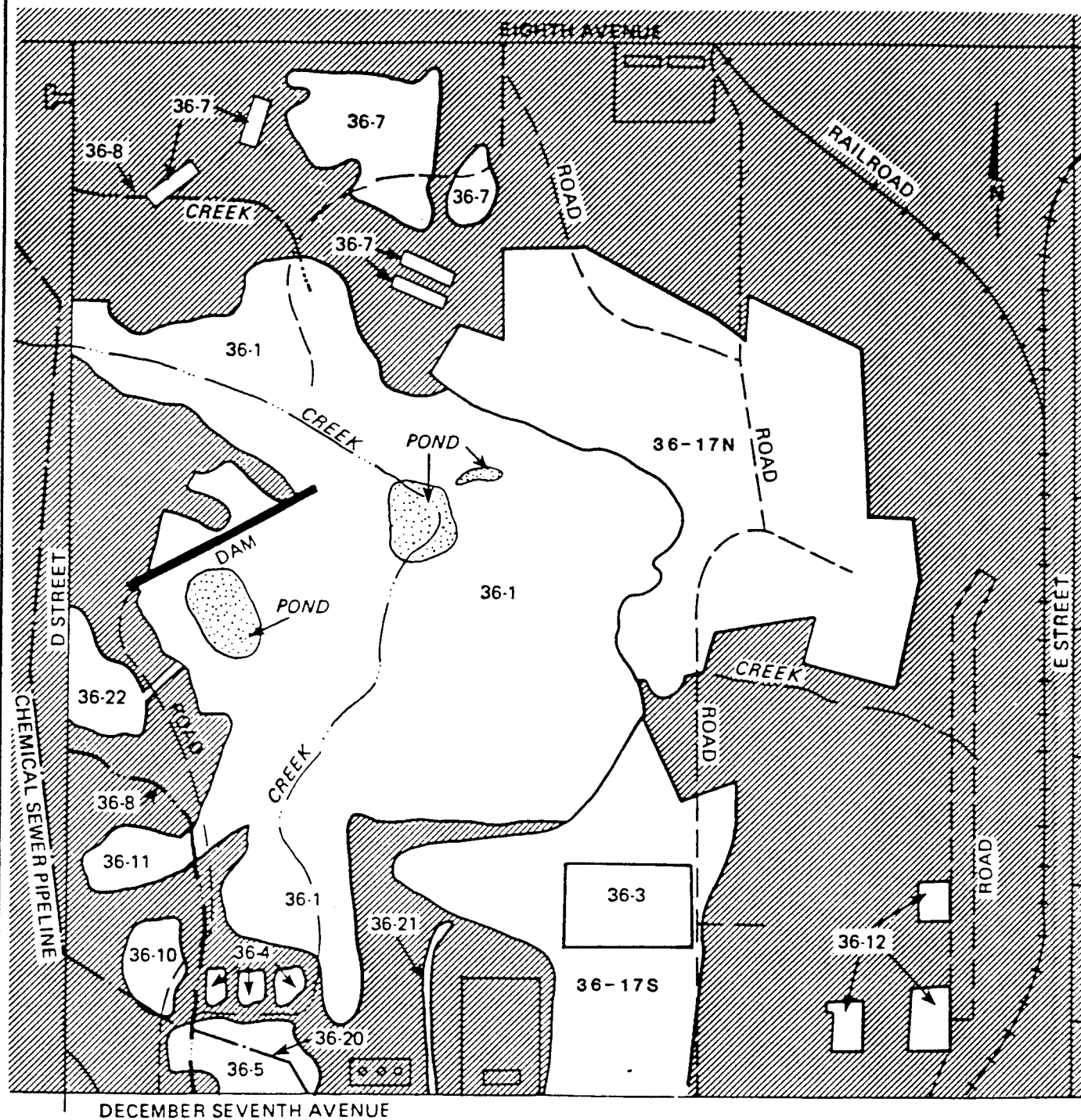


Figure 2.1-1
SECTION 36 CONTAMINATION SOURCES

water table is within 5 to 10 ft of the surface. The Denver Formation underlies the alluvium and consists of volcaniclastics, claystone, lignite, sand channels, lenticular sands, and a small continuous sand unit.

2.1.2 Source 36-3

Source 36-3, Shell insecticide pits, is located in the south-central portion of Section 36 as shown in Figure 2.1-1. This source is composed of a series of pits and trenches believed to have been used for the disposal of insecticides and pesticides such as aldrin, dieldrin, and endrin. Source 36-3 covers a maximum area of approximately 1.5 acres.

A study of aerial photographs taken between 1953 and 1975 indicates the following topographic information regarding Source 36-3 (Moloney 1982, p. 7-6, 7-7, 7-9, 7-11).

<u>Photo Date</u>	<u>Site Description</u>
1953	One long trench and an adjacent small trench are visible in Source 36-3.
1958	Four to eight additional trenches in the center of the site are apparent.
1962	Eight to twelve additional trenches are visible north and south of the existing trenches. An additional trench running diagonally to the northwest has been placed north of the site.
1975	Ten trenches and four pits have been added to the northern side of Source 36-3. A large area has been scraped clear on the southern side of Source 36-3. Five trenches and four pits are visible in the rest of the site. A drainage ditch to Basin A has been constructed.

The Shell insecticide pits are located on the southeastern edge of Basin A. The results of the Phase I program conducted by ESE have confirmed that Source 36-3 is underlain by alluvial material consisting of silty sands. Groundwater was observed at depths of 1 to 9 ft.

2.1.3 Source 36-17

Source 36-17, located in the eastern half of Section 36, is divided into northern (Source 36-17N) and southern (Source 36-17S) sectors extending almost the entire length of Section 36, as shown in Figure 2.1-1. Source 36-17 is characterized by a variety of disposal activities in numerous areas, with overlapping boundaries of imprecise history. Source 36-17 encompasses about 107 acres. Disposal activity in this area was first observed in a 1948 aerial photograph of Section 36. Activity in this source area continued from 1948 through 1975.

The center of Source 36-17N is situated on a bedrock high. Above the bedrock the alluvial section consists of 20 to 30 ft of silty sand. In Source 36-17S the alluvium is of similar thickness and composition, with the addition of interbedded clays.

2.2 CONTAMINANT CHARACTERISTICS

The discussion presented identifies the major contaminants in Sources 36-1, 36-3, and 36-17. The concentrations of contaminants were based on borings and testing conducted by ESE during the Phase I contamination survey (ESE 1987).

2.2.1 Source 36-1

Since 1943, a wide variety of organics, including pesticides and agents, have been introduced into Basin A. Metals and nonmetals are also present.

During the Phase I contamination survey, ESE drilled 62 borings yielding 119 samples. Table 2.2-1 presents a summary of analytical results for Source 36-1 from this Phase I program. Figure 2.2-1 depicts the location of samples containing concentrations of contaminants above their respective indicator ranges. Boring 36-1-3204A, in the southern portion of Source 36-1, has the highest concentration of organics, while Boring 36-1-3239A, at the center of Source 36-1, has the highest concentration of metals. The locations of these borings are marked by an "X" on Figure 2.2-1.

TABLE 2.2-1

SUMMARY OF ANALYTICAL RESULTS FOR SOURCE 36-1

Constituent	Samples Constituent Detected in		Range	Concentrations (µg/g)		
	Number of Samples	Percent of Samples		Mean	Standard Deviation	Detection Limit
<u>Volatiles (47 samples tested)</u>						
Chloroform (CHCl3)	1	2	0.8	BDL ^{1/}	BDL	0.3
Methylene Chloride (CH2Cl2)	2	4	1-2	1.5	BDL	0.3
<u>Semivolatiles (119 samples tested)</u>						
Aldrin	20	17	0.7-400	88	100	0.9
Dieldrin	36	30	0.4-700	50	100	0.3
p,p'-DDT	4	3	1-60	BDL	BDL	0.4
Endrin	13	11	1-200	40	50	0.7
Chlordane	19	16	5-400	80	100	1.0
p,p'-DDE	5	4	2-10	5	5	0.3
Diisopropylmethylphosphonate (DIMP)	17	14	0.6-10	5	3	0.5
Isodrin	13	11	0.7-200	20	50	0.3
Diathiane	2	2	0.6-0.8	BDL	BDL	0.3
Dicyclopentadiene (DCPD)	3	3	0.4-1	BDL	BDL	0.3
p-Chlorophenylmethylsulfide (PCPMS)	4	3	0.5-200	BDL	BDL	0.3
p-Chlorophenylmethylsulfoxide (PCPMSO)	1	1	6	BDL	BDL	0.4
p-Chlorophenylmethylsulfone (PCPMSO2)	8	7	0.4-6	BDL	BDL	0.3
Dibromochloropropane (DBCP)	3	3	0.007-0.11	BDL	BDL	0.005
Hexachlorocyclopentadiene (HCPD)	1	1	100	BDL	BDL	1.0
<u>Metals (119 samples tested)</u>						
Cadmium	32	27	0.60-9.4	1.7	1.7	0.9
Chromium	111	93	7-100	16	9.8	7.2
Copper	118	99	6-210	20	26	4.8
Lead	34	28	18-120	37	27	17
Zinc	97	81	16-200	56	29	16
Arsenic	87	73	5.5-1,100	79	140	4.7
Mercury	61	51	0.050-64	3.2	9.4	0.05

Source: ESE 1987

1/ BDL means below detection limits.

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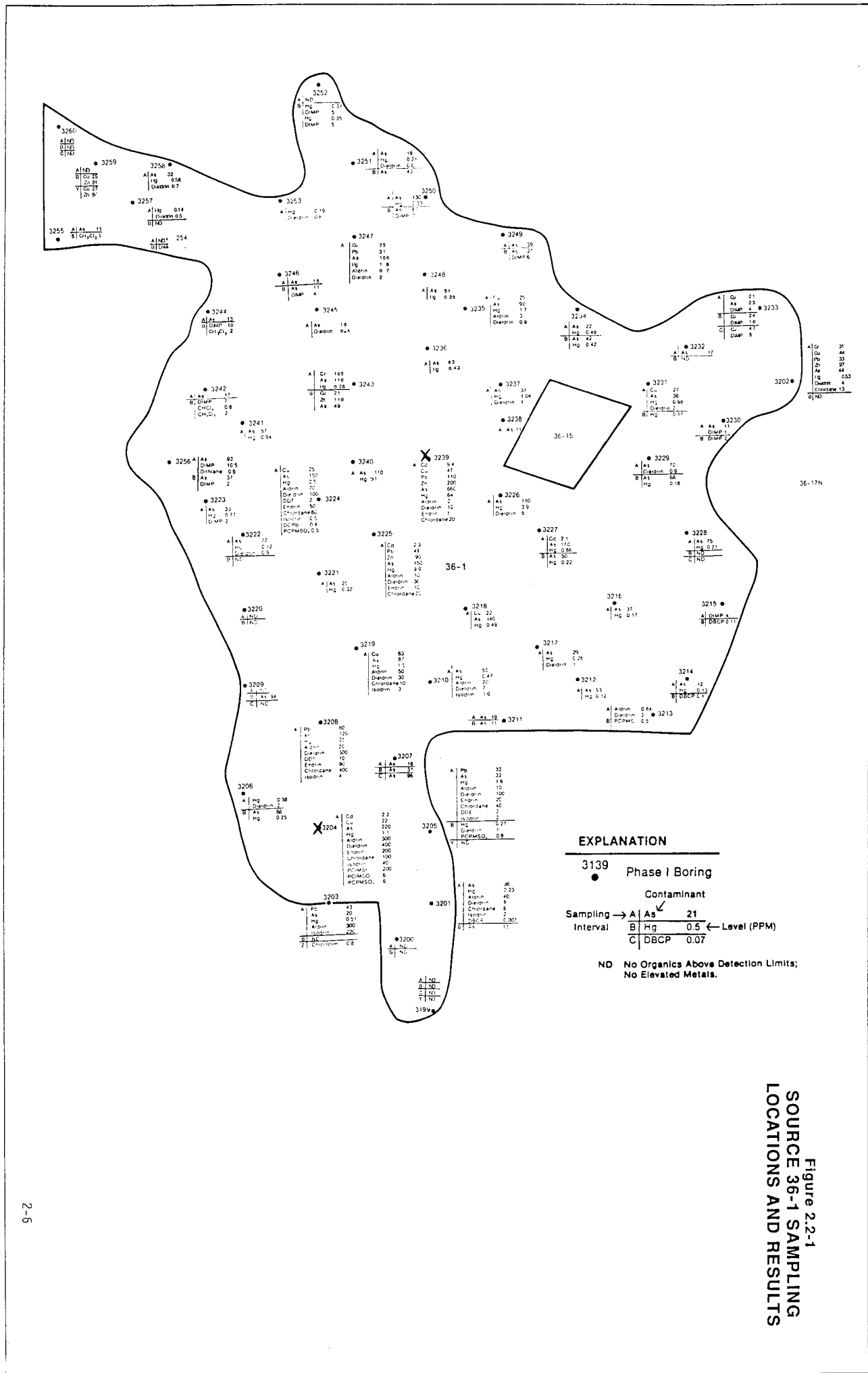


Figure 2.2-1
SOURCE 36-1 SAMPLING
LOCATIONS AND RESULTS

2.2.2 Source 36-3

Source 36-3 was a disposal area for insecticides. During Phase I investigations, ESE drilled eight borings yielding 16 soil samples. A descriptive summary of the analytical results of the sampling is presented in Table 2.2-2. The spatial relationship of chemicals found in concentrations above their respective indicator ranges is shown in Figure 2.2-2. The highest concentration of organics was present in Boring 36-3-3176C. The highest concentration of metals was present in Boring 36-3-3175A. The locations of these borings are marked by an "X" on Figure 2.2-2.

Of the numerous contaminants observed in samples from Source 36-3, organochlorine pesticides and dibromochloropropane (DBCP) are the most prevalent. These contaminants are widely distributed, occurring in seven surface interval samples as well as two middle and two deep interval samples. Individual concentrations range from slightly greater than detection limits to a maximum of 100 ppm for aldrin. Although four borings contain only surface contamination, the remaining four borings exhibit elevated concentrations in the deeper B and C sampling intervals. Similarly, elevated concentrations of dicyclopentadiene (DCPD), dibromochloropropane, and the organosulfur compounds occur in the deeper sampling intervals. Inorganic constituents were observed at elevated concentrations only in surface interval samples, and their areal distribution appears sporadic.

The relationship between deep and shallow soil contamination for Source 36-3 is unclear based on the present sample distribution. No clear trends in areal or depth distribution are evident. Disposal of wastes in discrete trenches, followed by subsequent closure and reworking of surface soils, could result in the observed variability of concentrations with depth.

2.2.3 Source 36-17

Under the Phase I program, ESE drilled 46 borings to determine the extent of contamination at this site. The sampling program at this source consisted

TABLE 2.2-2

SUMMARY OF ANALYTICAL RESULTS FOR SOURCE 36-3

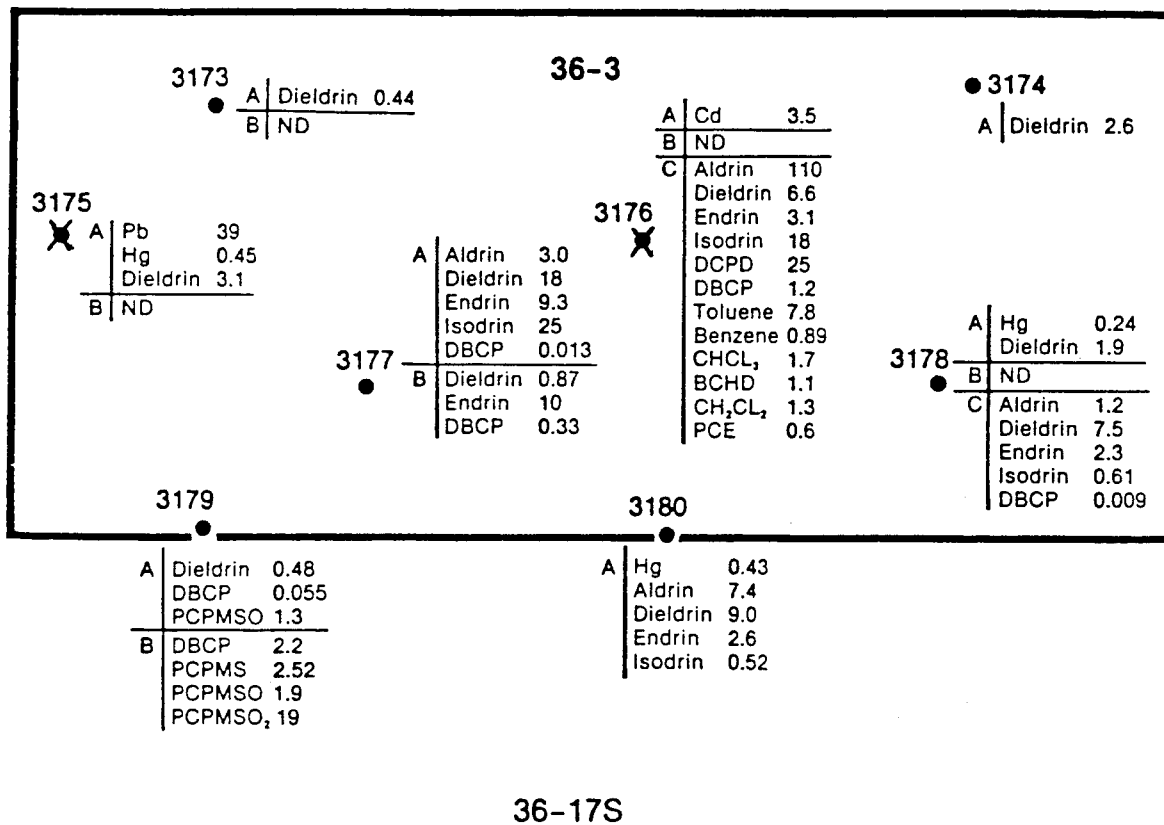
Constituent	Samples Constituent Detected in		Range	Concentrations (µg/g)		
	Number of Samples	Percent of Samples		Mean	Standard Deviation	Detection Limit
Volatiles (2 samples tested)						
Chloroform (CHCl3)	1	50	2.0	BDL ^{1/}	BDL	0.3
Bicycloheptadiene (BCHD)	1	50	1.0	BDL	BDL	0.3
Methylene Chloride (CH2Cl2)	1	50	1.0	BDL	BDL	0.3
Benzene	1	50	0.9	BDL	BDL	0.3
Tetrachloroethene (PCE)	1	50	0.6	BDL	BDL	0.3
Toluene	1	50	8.0	BDL	BDL	0.3
Semivolatiles (16 samples tested)						
Aldrin	4	25	1.0-100	BDL	BDL	0.9
Dieldrin	10	62	0.4-20	5	6	0.3
Endrin	5	31	2-10	6	4	0.7
Isodrin	4	25	0.5-20	BDL	BDL	0.3
Dicyclopentadiene (DCPD)	1	6	20	BDL	BDL	0.3
PCPMS	1	6	2	BDL	BDL	0.3
PCPMSO	2	12	2	BDL	BDL	0.4
PCPMSO2	1	6	20	BDL	BDL	0.3
Metals (16 samples tested)						
Cadmium	7	44	1.0-3.5	5	0.91	0.9
Chromium	13	81	9-21	13	3.3	7.2
Copper	13	81	6-29	9.8	6	4.8
Lead	8	50	17-68	29	17	17
Zinc	16	100	16-65	38	13	16
Arsenic	5	31	4.9-5.3	5.1	0.14	4.7
Mercury	10	62	0.06-0.45	0.16	0.16	0.05

Source: ESE 1987

^{1/} BDL means below detection limits.

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EXPLANATION

3139 Phase I Boring

Contaminant

Sampling Interval → A | As 21
 B | Hg 0.5
 C | DBCP 0.07

← Level (PPM)

ND No Organics Above Detection Limits;
 No Elevated Metals.

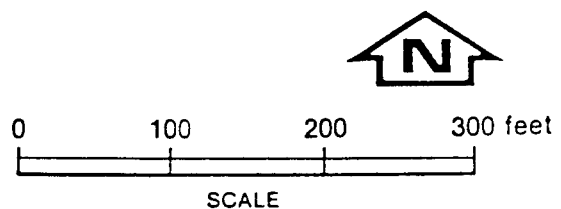


Figure 2.2-2

SOURCE 36-3 SAMPLING LOCATIONS AND RESULTS

of 99 samples. A summary of the analytical results of the sampling is presented in Table 2.2-3. Sample locations containing concentrations above indicator ranges are shown in Figures 2.2-3 and 2.2-4. For Source 36-17N, the highest concentrations of organics and metals were present in Borings 36-17-3065A and 36-17-3086Y, respectively. The locations of these borings are marked by an "X" on Figure 2.2-4.

Analysis of Source 36-17 Phase I soil samples indicated the presence of organochlorine pesticides including aldrin, dieldrin, endrin, diisopropylmethylphosphonate (DIMP), chlordane, and dibromochloropropane; organosulfur compounds; and elevated levels of metal concentrations. Concentrations ranged from slightly greater than their respective detection limits to very high levels in isolated areas. All samples taken from this source were tested for the presence of agents by the RMA laboratory, which analyzed a composite of each day's samples. If positive readings were found, individual samples from each boring were analyzed to identify the sample location. Positive readings for mustard were found in Borings 3900, 3092, 3093, and 3094. Samples from these borings were consequently not analyzed for other constituents.

2.3 FIELD SAMPLING PROGRAM

The sampling for the Expansion Program involved the collection of contaminated soil samples from Sources 36-1, 36-3, and 36-17 in sufficient quantities for laboratory analyses and incineration/thermal treatment. Sampling operations conformed to the guidelines and procedures established for Task 2 (i.e., "RMA Procedures Manual, Volume I: Sampling" and "Sampling and Chemical Analysis Quality Assurance Program for U.S. Army Toxic and Hazardous Materials Agency").

For the incineration studies, the samples with the highest concentration of organics were collected to obtain a measurable destruction and removal efficiency using gas chromatography/mass spectrometry (GC/MS).

TABLE 2.2-3

SUMMARY OF ANALYTICAL RESULTS FOR SOURCE 36-17

Constituent	Samples Constituent Detected in		Concentrations (µg/g)			
	Number of Samples	Percent of Samples	Range	Mean	Standard Deviation	Detection Limit
<u>Volatiles (7 samples tested)</u>						
None Detected	BDL ^{1/}	BDL	BDL	BDL	BDL	0.3
<u>Semivolatiles (99 samples tested)</u>						
Aldrin	3	3	1-1,000	BDL	BDL	0.9
Dieldrin	6	6	0.3-500	80	200	0.3
Endrin	1	1	0.9	BDL	BDL	0.7
Chlordane	2	2	60-70	BDL	BDL	1.0
p,p'-DDE	1	1	3	BDL	BDL	0.3
Diisopropylmethylphosphonate (DIMP)	5	5	0.7-4	2	2	0.5
p-Chlorophenylmethylsulfide (PCPMS)	1	1	0.7	BDL	BDL	0.3
Dibromochloropropane (DBCP)	6	6	0.012-0.44	0.12	0.16	0.005
<u>Metals (99 samples tested)</u>						
Cadmium	12	12	0.70-13	3.3	4.1	0.9
Chromium	83	84	7.0-1,400	30	160	7.2
Copper	95	96	5.0-660	21	68	4.8
Lead	25	25	17-7,100	310	1,400	17
Zinc	88	89	24-12,000	200	1,300	16
Arsenic	23	23	4.7-29	10	7.1	4.7
Mercury	26	26	0.050-1.2	0.18	0.24	0.05

Source: ESE 1987

^{1/} BDL means below detection limits.

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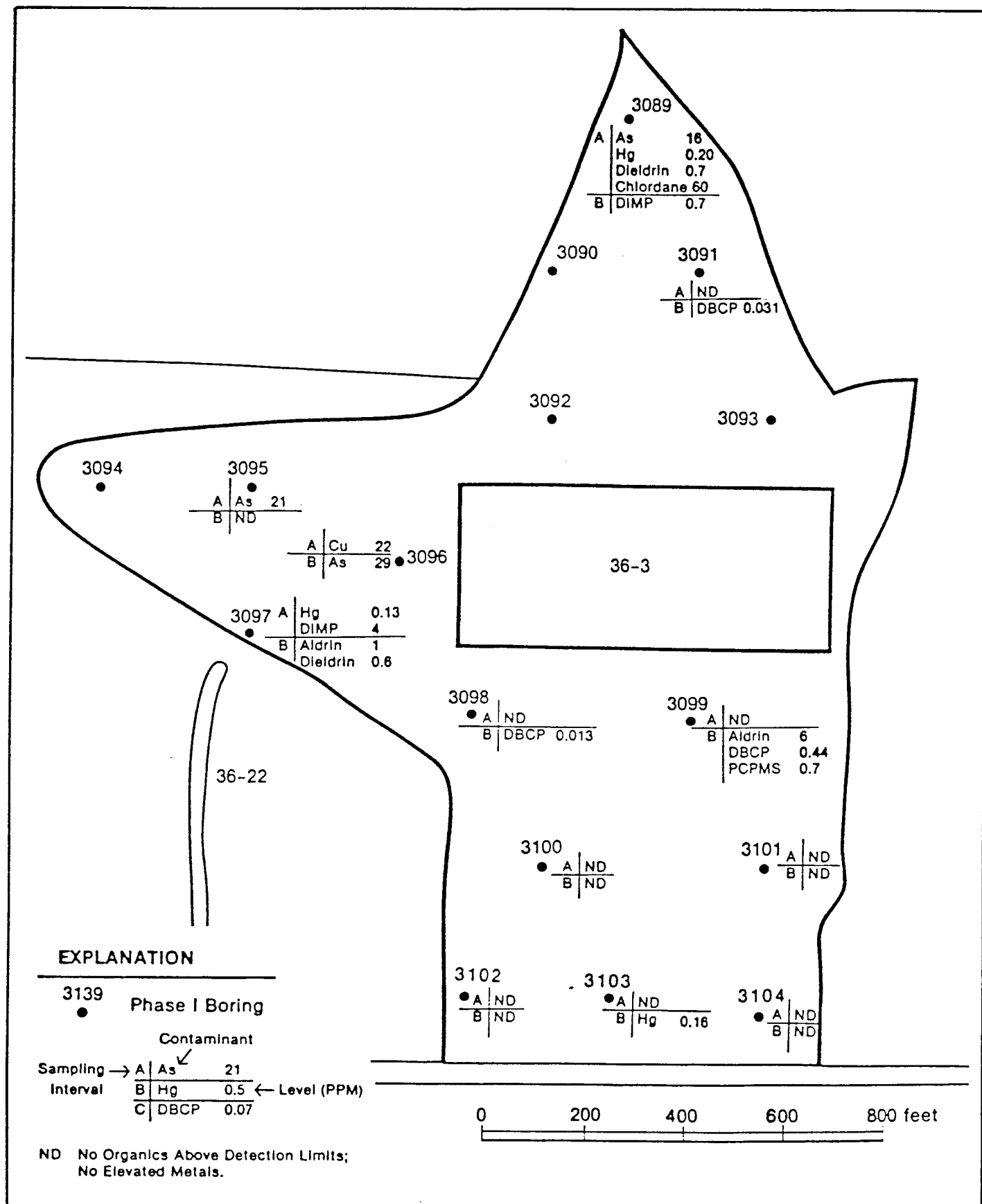
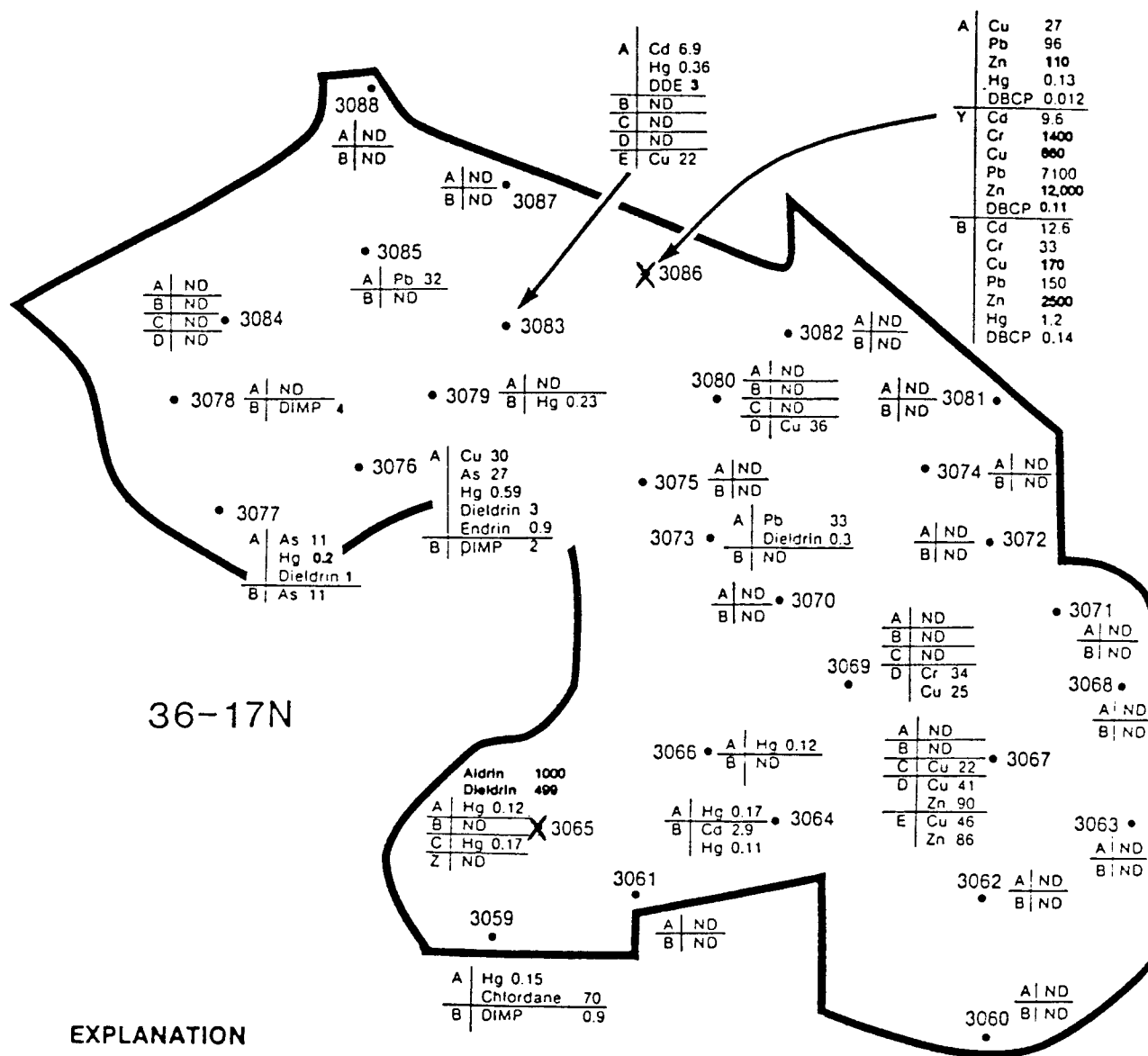


Figure 2.2-3
SOURCE 36-17S SAMPLING LOCATIONS AND RESULTS



EXPLANATION

3139	Phase I Boring
Sampling Interval	Contaminant
A	As 21
B	Hg 0.5
C	DBCP 0.07

← Level (PPM)

ND No Organics Above Detection Limits;
No Elevated Metals.

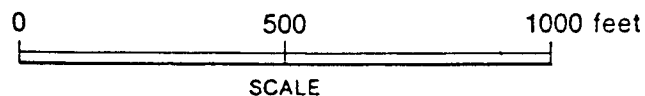


Figure 2.2-4
SOURCE 36-17N SAMPLING LOCATIONS AND RESULTS

The following is a discussion of the locations where soil samples were obtained for each of the three sources. A brief discussion of the materials' characteristics is also given.

2.3.1 Source 36-1

Under the Phase I site investigation program, ESE drilled 62 borings and analyzed 119 samples. Of these analyses, a soil sample from Boring 3204A (see Figure 2.2-1) was found to contain the highest concentrations of organics (i.e., aldrin, dieldrin, chlordane, isodrin, endrin, chlorophenylmethylsulfide [PCPMS], chlorophenylmethylsulfoxide [PCPMSO], and chlorophenylmethylsulfone [PCPMSO₂]). The highest concentrations of organics were limited to the sample collected from the 0- to 1-ft interval. Similarly, soil samples taken from Boring 3239A (0- to 1-ft interval) exhibited the highest concentrations of the metals cadmium, chromium, copper, lead, zinc, arsenic, and mercury. Therefore, for the purpose of this Expansion Program, approximately 15-kilograms of soil sample were excavated to a depth of 1 ft using hand shovels from each location close to Borings 3204 and 3239.

2.3.2 Source 36-3

ESE drilled eight borings and analyzed 16 soil samples for Source 36-3 under the Phase I program. A sample taken from Boring 3176C (9- to 10-ft interval) exhibited the highest concentrations of organic contaminants. A sample taken from Boring 3175A (0- to 1-ft interval) exhibited the highest concentrations of metal contaminants. Therefore, for this Expansion Program, 15-kilogram soil samples from both locations were collected. (For locations of Borings 3175A and 3176C see Figure 2.2-2.)

2.3.3 Source 36-17

In this area, ESE drilled 46 borings and tested 99 samples for contamination assessment. The sample from Boring 3065A (1- to 2-ft interval) exhibited the highest concentrations of organics. The sample from Boring 3086Y

exhibited the highest concentrations of metals (2- to 2.5-ft interval). For this Expansion Program, 15-kilogram soil samples from both locations were collected.

2.4 LABORATORY PROGRAM

The objectives of this investigation were to show that the previous optimum conditions for the primary reactor and afterburner could be applied to Section 36 soils. Also, an investigation was made into PIC formation, residence time, low temperature conditions in the primary chamber, and metal partitioning. EP Toxicity and TCLP analyses were conducted to determine if the residue following thermal treatment was hazardous.

To meet these objectives, the laboratory program for the Section 36 samples involved the following experiments:

- 1) Organic Volatilization in a Muffle Furnace;
- 2) Metal Volatilization in a Muffle Furnace;
- 3) Volatilization in the Primary Reactor;
- 4) Temperature Profile in the Primary Reactor;
- 5) Basin A Bench-Scale Test Burn; and
- 6) EP Toxicity and TCLP Analyses.

A description of the incinerator systems employed, and the purpose, procedures, and conditions for each experiment are presented in Chapter 3.0.

As indicated in the previous section, samples were obtained adjacent to Borings 36-1-3204A, 36-1-3239A, 36-3-3175A, 36-3-3176C, 36-17-3065A, and 36-17-3086Y. Of these samples, concentrations from ESE indicated that the sample from Boring 36-1-3204A would be ideal for organic volatilization and bench-scale experiments, while the sample from Borings 36-1-3239A and 36-17-3086Y would be ideal for metal volatilization experiments. The EP Toxicity and TCLP analyses were performed on all of the boring samples.

Feedstock analyses for organics and metals were performed to verify the selection of the samples for the various aspects of the experimental program. The results of these analyses are presented in Table 2.4-1 (values

TABLE 2.4-1
CONCENTRATION OF METALS AND ORGANICS FROM THE BORINGS
USED IN THE LABORATORY ANALYSIS ($\mu\text{g/g}$)^{1/}

	36-1-3204A	36-1-3239A	36-17-3086Y	36-17-3065A	Range for Basin F Samples
<u>Metals</u>					
Arsenic	313.0	213.0	60.0	BDL ^{2/}	NA ^{3/}
Cadmium	<3.7	3.0	15.0	BDL	NA
Chromium	12.0	9.5	76.0	BDL	NA
Copper	16.0	24.0	1,030.0	BDL	NA
Lead	15.0	59.0	406.0	(0.1)	NA
Mercury	1.4	24.0	<0.1	BDL	NA
Zinc	32.0	70.0	1,150.0	BDL	NA
<u>Organics</u>					
Aldrin	32,000-85,000	(2)	BDL	BDL	1,700-3,900
Dieldrin	19,000-76,000	(12)	BDL	BDL	1,000-2,800
Endrin	2,600-7,800	(5)	BDL	BDL	170-610
Chlordane	6,100-7,700	(20)	BDL	BDL	<2
Isodrin	2,400-6,300	BDL	BDL	BDL	89-300
PCPMS	3,200-8,300	BDL	BDL	BDL	1,400-2,700
DCPD	BDL	BDL	BDL	BDL	59-240

^{1/} Concentrations are from the Ebasco laboratory program except for values in parentheses, which are from ESE (1987).

^{2/} BDL means below detection limit.

^{3/} The concentrations of metals were not determined for Basin F samples.

Note: Although samples from Borings 36-3-3175A and 36-3-3176C were collected, the material was never used since it had very low concentrations of both metals and organics.

in parentheses are from ESE [1987]). Also presented are the ranges of concentrations of organics for the Basin F samples previously utilized (metals analyses were not conducted on Basin F samples). The results indicate that the sample from Boring 36-1-3204A was high in organics and that the samples from Borings 36-1-3239A and 36-17-3086Y were high in metals. The table also indicates that the sample from Boring 36-1-3204A had a much higher concentration of organics when compared to ESE (1987) data and to the sample used for the Basin F bench-scale runs. Also, the Basin F samples had essentially zero (less than 2 $\mu\text{g/g}$) chlordane, while the sample from Boring 36-1-3204A had between 6,100 and 7,700 $\mu\text{g/g}$ of chlordane.

3.0 INCINERATION TEST APPARATUS AND PROCEDURES

This chapter summarizes the laboratory apparatus and procedures used to conduct the following experiments on Section 36 soil samples:

- 1) Organic Volatilization Experiments
- 2) Metal Volatilization Experiments
- 3) Volatilization in the Primary Reactor (Trapping Experiment)
- 4) Temperature in Bench-Scale Unit
- 5) Bench-Scale Test Burns
- 6) EP Toxicity and TCLP Analyses

3.1 INCINERATION SYSTEMS

To conduct the experiments under this program, two different incineration systems were utilized: the bench-scale incinerator system and the muffle furnace system. The bench-scale system provided a means of volatilizing the organics and determining what conditions were required in the afterburner to destroy the volatilized organics while minimizing PICs. The muffle furnace was used to determine what temperatures and residence times were required in the primary chamber to volatilize the organics in the soil matrix. A brief description of both systems is provided below. A detailed description of the bench-scale incineration system can be found in Chapter 3.0 of the Basin F Laboratory Report (Ebasco 1988).

3.1.1 Bench-Scale Incineration System

The laboratory bench-scale unit was previously described in detail in the Basin F Laboratory Report. However, a brief description of applicable experiments is presented here.

A schematic representation of the laboratory bench-scale unit is presented in Figure 3.1-1. As previously described, the incinerator used electric resistance heating and as such was a nonflame mode incinerator. The

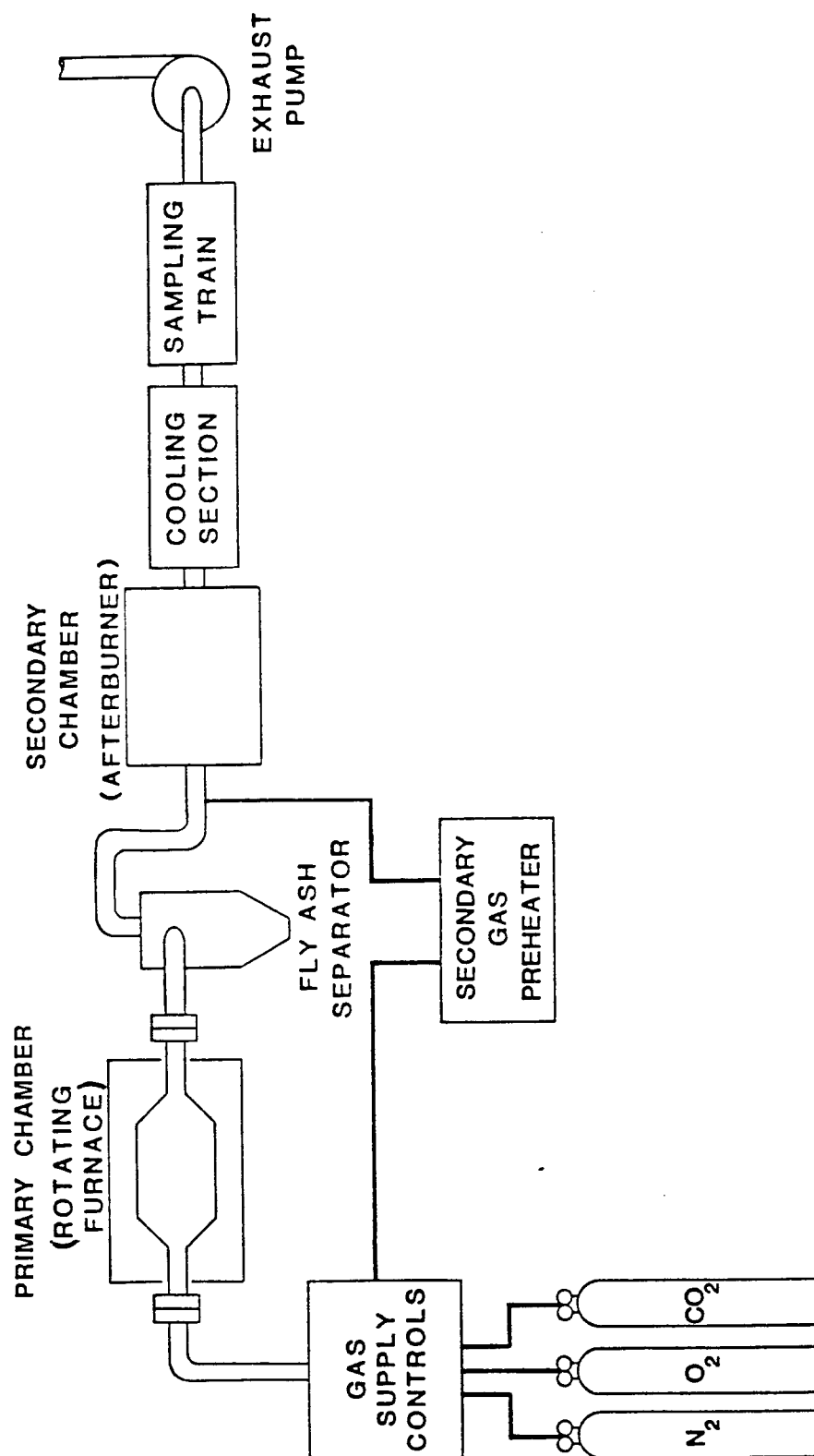


FIGURE 3.1-1
LABORATORY BENCH-SCALE INCINERATION UNIT

dimensions of the rotating furnace are presented in Figure 3.1-2. The cylindrical part of the furnace measures 200 mm (7.9 inches) long by 130 mm (5.1 inches) in diameter. Sample sizes used were 300 grams. The purposes of the major system components are described below.

Primary Chamber: The purpose of the primary chamber was to volatilize the organics from the soil matrix at various temperatures, reaction times, and oxygen levels.

Afterburner: The purpose of the afterburner was to destroy the volatilized organics while minimizing the formation of PICs. The afterburner was constructed using a pottery kiln. Quartz tubes of various lengths were used to vary the gas residence time (2 and 5 seconds).

Gas Preheater: The gas preheater was used to preheat the combustion air for the afterburner to the exit temperature of the primary chamber. The purpose of air preheating was to minimize gas cooling and thus the formation of condensation products.

Gas Cooling and Scrubbing: The gas cooling and scrubbing system was used to cool the gas and to remove particulates, acid gases, and metals.

3.1.2 Muffle Furnace System

The muffle furnace system was used to conduct experiments to replicate the behavior of the primary reactor of the bench-scale unit. The furnace used was the Fisher Model 495A Ashing Furnace. The performance characteristics of the furnace are presented in Table 3.1-1. Of importance to organic volatilization experiments was the rapid heatup rate of the furnace of between 10°C to 35°C/minute. This rapid heatup rate more readily approaches the actual heatup rate in flame mode pilot or full-scale incinerators than that possible of the primary reactor of the bench-scale incinerator. Actual heatup rates (at a 230 volt input) are presented below.

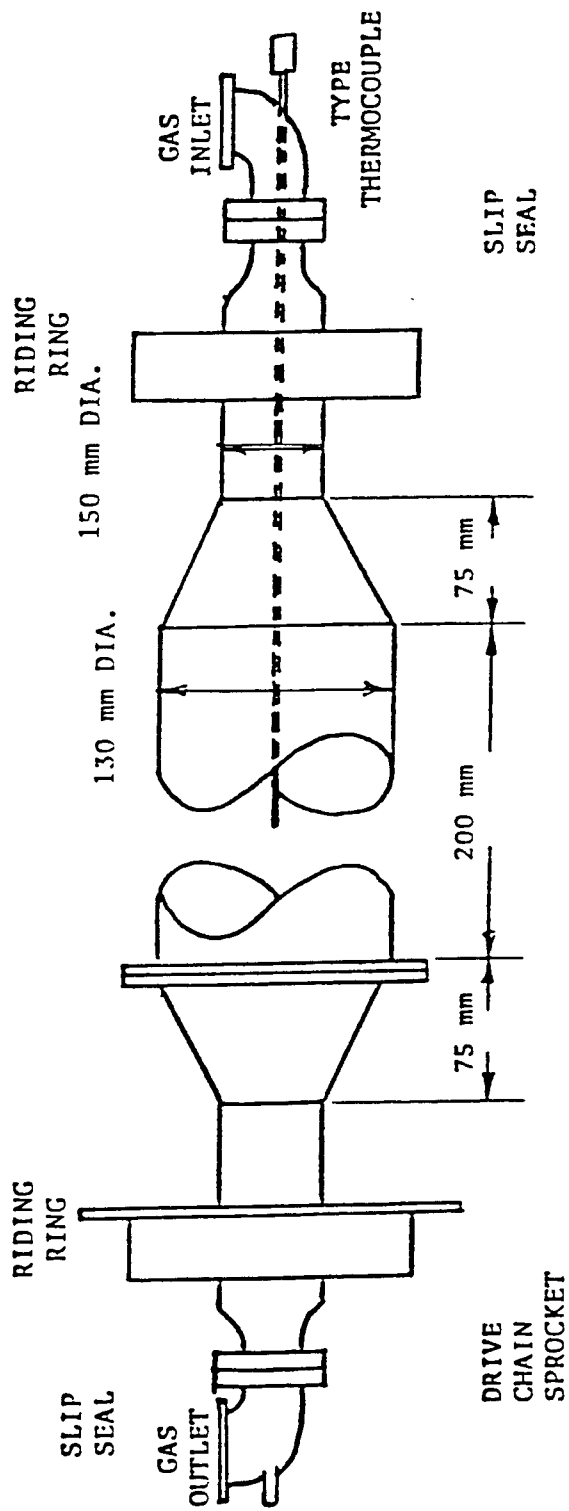


FIGURE 3.1-2
ROTATING TUBE UNIT DIMENSIONS

TABLE 3.1-1
PERFORMANCE CHARACTERISTICS OF THE ASHING FURNACE

Factor	Characteristic
Temperature Range	Ambient to 1,100°C
Set Point Accuracy	Less than $\pm 5^{\circ}\text{C}$
Rise Time	Less than 30 minutes to reach 700°C
Cool Down	Less than 30 minutes from 1,100°C to 200°C
Linear Heating Rates	Variable between 1°C and 35°C/min
Linear Cooling Rates	Variable between 1°C and 14°C/min
Atmospheric Changes	3/minute (fan on)
Chamber Dimensions	6.5 x 4.75 x 10.75 inches

<u>Temperature Range, °C</u>	<u>Maximum Liner Heating Rate - Fan Off, °C/min</u>	<u>Time to Temperature Fan Off, min</u>
25 - 400	31	12
400 - 750	27	25
750 - 930	16	36
930 - 1,100	10	53

The furnace is programmable in both the heating and cooling modes. The heatup and cooling programs can each automatically increase or decrease the temperature to up to three different levels and hold at each temperature for a predetermined time.

The unit is equipped with a gas inlet port and a fan damper to facilitate operating the furnace with inert atmospheres or various combustion atmospheres using a variety of cylinder gases. The flow rate of inert gas required to achieve a given oxygen level is presented below.

<u>Flow Rate, Liters/min</u>	<u>Approximate Percent of Oxygen</u>	<u>Atmosphere Changes/ min</u>
0	21	0
10	8	2
20	5	3.7
30	2	5.5

3.2 ORGANIC VOLATILIZATION EXPERIMENTS

Organic volatilization studies were conducted to determine the conditions necessary to volatilize 99.99 percent of the organic constituents present in Basin A soils into the vapor phase. The amount of organics in the vapor phase was determined by analyzing for the 22 "target" compounds in both the feedstock and residue following thermal treatment. The chemical structure of these compounds is presented in Appendix A. The organic volatilization study was conducted on a Basin A sample from Boring 36-1-3204A because this was the only sample that had a sufficient concentration of a variety of organics. It is important to realize that the achievement of a 99.99 percent volatilization does not mean that 99.99 percent of the organics are destroyed. It only means that the organics are removed to the

off-gases to that level. As a result, a destruction and removal efficiency was not calculated. A description of the conditions and procedure for the organic volatilization studies is presented below.

3.2.1 Test Conditions

The organic volatilization procedure involved the controlled heating of various samples in a muffle furnace at different temperatures and residence times. The muffle furnace atmosphere was kept at 11.5 percent oxygen, a practical lower limit for the device. A broad range of furnace conditions was possible. However, the following test conditions were selected:

<u>Temperature, °C</u>	<u>Residence Time, min</u>
200	22.5 and 45
400	15 and 30
500	40
600	7.5 and 22.5
800	7.5 and 15

These values were selected for the following reasons:

- 1) The values selected account for the expected range in primary reactor temperatures and residence times.
- 2) Interpolation to other conditions could be easily accomplished.

Because of the high moisture content of the samples, it was not possible to preheat the muffle furnace to the above stated temperatures. This mode of operation would cause a rapid flash of steam that could prove dangerous to the operator and could also release toxic contaminants. As a result, the following temperature programming was used:

<u>Temperature Range, °C</u>	<u>Heatup Rate, °C/min</u>	<u>Time at Temperature, min</u>
25 - 105	5	60
106 - 250	10	15
251 - 900	30	Residence Time

A breakdown of the above temperature program for each of the conditions described above is presented in Table 3.2-1. An assumption was made that organic volatilization at 105°C was insignificant (heatup time 16 minutes and residence time 60 minutes). This assumption was based on the fact that most pesticides are stable at or below 120°C. For example, aldrin and malathion only lose about 2 to 3 percent of their weight at 100°C with no exothermic reactions occurring until 120°C (Ferguson et al. 1975). This same trend has been observed for other pesticides, including toxaphene, atrazine, captan, zineb, mirex, and picloram. The effect of soil moisture stripping of organics was not considered. However, if it occurred, it would be equal for all samples, thus a consistent baseline would still be maintained.

3.2.2 Test Procedure

The procedure involved the thermal treatment of the samples in a muffle furnace at an 11.5 percent oxygen level. Sufficient sample was used to achieve the necessary detection limits to determine a 99.99 percent volatilization. The sample was weighed and placed in a tared, low profile crucible. Low profile crucibles were used to maximize the surface area exposed to the radiant energy of the muffle furnace chamber. The sample was then placed into the muffle furnace and temperature was increased to 105°C and held for 60 minutes. The temperature was then raised to the appropriate test condition. Following thermal treatment, the crucible was removed, placed in a desiccator to cool, and weighed to calculate weight loss. The thermally treated sample was then extracted for GC/MS analysis to determine the concentration of the target compounds. This procedure was repeated for each of the nine test conditions indicated above. A GC/MS analysis for target compounds was also done on the homogenized feedstock.

3.3 METAL VOLATILIZATION EXPERIMENTS

Metal volatilization studies were conducted to determine which metals left the soil matrix as a function of temperature, residence time, and atmosphere. A description of the conditions and procedure is presented below.

TABLE 3.2-1
HEATUP AND RESIDENCE TIMES FOR SELECTED TESTS

Test Conditions			
Residence Temperature (°C)	Residence Time ^{1/} (min)	Heatup Time to Temperature ^{2/} (min)	Total Time Above 200°C (min)
200	22.5	9.5	32.0
200	45.0	9.5	54.5
400	15.0	19.5	34.5
400	30.0	19.5	49.5
500	40.0	23.0	63.0
600	7.5	26.0	33.5
600	22.5	26.0	48.5
800	7.5	33.0	40.5
800	15.0	33.0	48.0

^{1/} Time at temperature once the test temperature was achieved.

^{2/} Heatup times do not include the heatup time and residence time at 105°C.

3.3.1 Test Conditions

These experiments were conducted in the muffle furnace. The metals tested for included arsenic, cadmium, chromium, copper, lead, mercury, and zinc, all of which have been identified at Rocky Mountain Arsenal. The boiling points (temperature of sublimation for arsenic) for each of these elements are shown below:

Arsenic	615°C
Cadmium	767°C
Chromium	2,200°C
Copper	2,324°C
Lead	1,620°C
Mercury	357°C
Zinc	907°C

The metal volatilization studies were conducted at the following temperatures: 200°C, 400°C, 600°C, and 1,000°C, as well as the following residence times: 15, 30, and 45 minutes. The test conditions are presented in Table 3.3-1. The tests were conducted on samples from Borings 36-1-3239A and 36-17-3086A in both an oxidizing (21 percent oxygen) and reducing (0 percent oxygen) atmosphere. As a result, 40 runs were conducted. The above samples were selected because they had the highest concentration of metals.

3.3.2 Test Procedure

The procedure for the metal volatilization experiments was identical to that previously described for the organic volatilization experiments except for the sample size, temperatures, and residence times. The samples were digested and analyzed for cadmium, chromium, copper, lead, and zinc by inductively coupled plasma (ICP); by graphite furnace atomic absorption (GFAA) for arsenic; and by cold vapor atomic absorption (CVAA) for mercury.

3.4 VOLATILIZATION IN THE PRIMARY REACTOR (TRAPPING EXPERIMENT)

The test results of the Basin F laboratory investigation indicated that the target compounds present in the soil could be destroyed to a 99.99 percent

TABLE 3.3-1
MATRIX FOR METAL VOLATILIZATION TESTS IN THE MUFFLE FURNACE

Temperature (°C)	Residence Time (minutes)		
	15	30	45
200		X	X
400		X	X
600		X	X
800	X	X	
1,000	X	X	

Note: "X" indicates analyses to be performed on residue samples derived at those conditions.

DRE at a primary chamber temperature of 900°C, an afterburner temperature of 1,200°C, an afterburner residence time of 2 seconds, and a 7 percent oxygen level. However, the degree to which compounds volatilize in the primary reactor as a function of temperature was not known. As a result, an experiment was devised using Basin A soil to qualitatively measure the rate of volatilization of the target compounds.

3.4.1 Test Conditions

In order to measure the rate of volatilization, the primary chamber was decoupled from the afterburner. The unit was to be increased in temperature in a step-wise fashion collecting the volatilized organics after each step. However, because of technical problems, the primary chamber could not be adapted to this use. Specific problems discovered in using the primary chamber are indicated below:

- o Plugging of lines;
- o Difficulty in adapting the outlet to the trapping device;
- o Difficulty in achieving the desired level of temperature control;
and
- o Expense of revamping the system to conduct the trapping experiment.

Because of initial failures using the primary chamber, the muffle furnace was used as the primary reactor.

3.4.2 Test Procedure

Research has shown that the majority of the organics present in the soil matrix volatilize below 400°C (Ferguson et al. 1975). As a result, the trapping experiment was conducted at temperatures below 400°C.

The procedure began with the placement of a sample into the muffle furnace. The unit was then heated to 100°C as rapidly as possible, and remained at 100°C for 15 minutes. The same procedure was followed for temperature settings of 200°C, 300°C, and 400°C. During the heatup time to a given

temperature and the residence time at a given temperature, the organics in the off-gases were trapped on activated carbon and tenax adsorbents. Four different traps were used corresponding to the four temperatures utilized. A schematic representation of the experimental setup is shown in Figure 3.4-1. Three-way valves were used to direct the off-gas flows.

It was originally planned to use a Basin A sample for the trapping experiment. However, with the Basin A sample, insufficient material was collected in the traps. As a result, Basin F soil samples were utilized. The experiment was conducted at 0 percent oxygen under a nitrogen atmosphere. The traps were then removed, extracted, and analyzed by GC/MS for the target organic compounds. The residue from the muffle furnace was also analyzed by GC/MS for the target organic compounds.

3.5 TEMPERATURE IN BENCH-SCALE UNIT

During the Basin F test burns, the analysis for PICs revealed the presence of alcohols, carbonyls, and esters. These compounds are typical oxidation products of hydrocarbons at temperatures below 450°C. It was then suspected that the observed oxidation products were forming in the cooler regions of the primary chamber of the bench-scale unit. However, these compounds could also be formed in cool regions of the soil and in the piping following the afterburner. Subsequently, lines beyond the afterburner were heat traced and insulated. To evaluate cold spots in the primary reactor, a temperature profile was conducted.

In order to identify the possibility of low temperatures in the primary reactor, a temperature profile across the horizontal axis of the primary reactor was conducted by pulling a properly marked (for measurement of position on the horizontal axis) thermocouple along the primary chamber's horizontal axis. This procedure was done at the following temperatures: 400°C, 650°C, 800°C, and 900°C. A schematic representation of the primary chamber components is provided in Figure 3.5-1. The zero position point was at the midpoint of the reactor. Points measured were 19 mm (0.75 inches) apart until a distance of 730 mm (29 inches) was covered, at which point the probe exited the gas infeed end of the primary chamber.

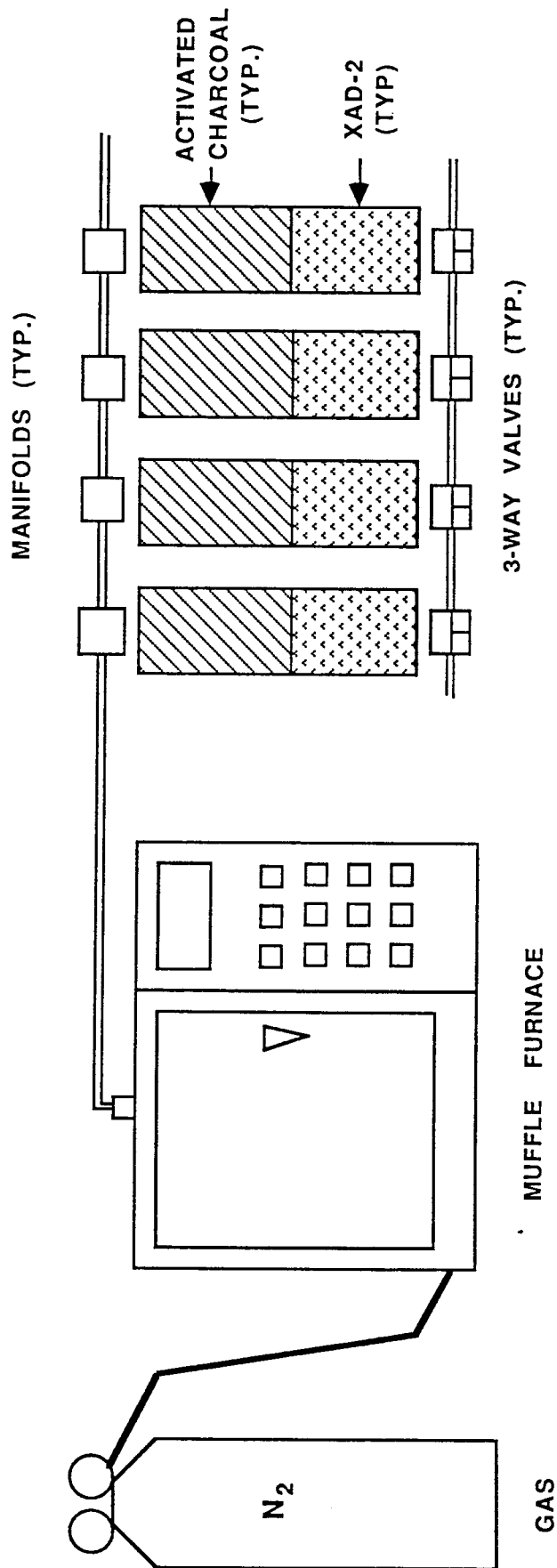


FIGURE 3.4-1

SCHEMATIC REPRESENTATION OF THE EXPERIMENTAL SETUP FOR THE ORGANIC VOLATILIZATION EXPERIMENT UTILIZING A DECOUPLED AFTERBURNER

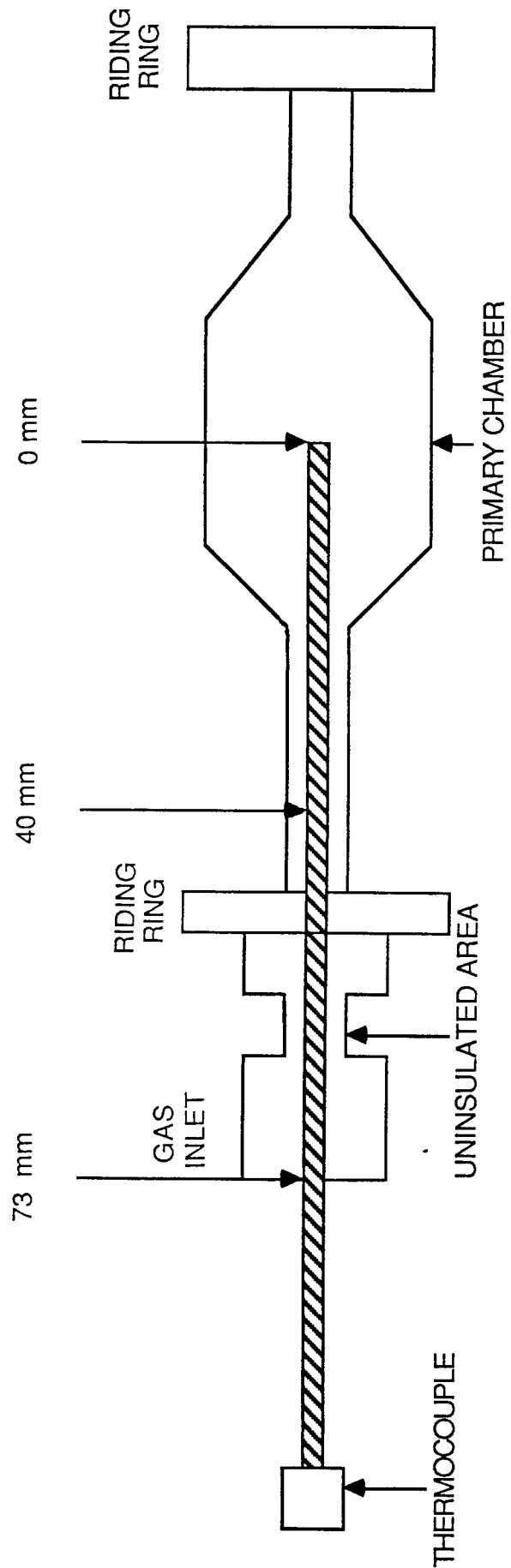


FIGURE 3.5-1
CUT AWAY VIEW SHOWING THERMOCOUPLE'S
RELATIVE POSITION

3.6 BENCH-SCALE TEST BURNS

Previous bench-scale experiments conducted on Basin F soil samples indicated that a DRE of 99.99 percent was achieved on the target compounds while minimizing the PICs at the conditions summarized below:

Primary Chamber Temperature	900°C
Afterburner Temperature	1,200°C
Afterburner Residence Time	2 seconds
Oxygen Level	7 percent

Based on the success of the Basin F experiments, it was decided to extend the experiments to Section 36 soil samples, which include Basin A. The two major goals of this investigation are summarized below:

- o The first goal was to determine if the target compounds in the Section 36 samples could be destroyed to a 99.99 percent DRE.
- o The second goal was to evaluate the feasibility of operating the primary chamber at low temperature conditions (450°C to 600°C), while maintaining the afterburner conditions at a temperature of 1,200°C, with a gas residence time of 2 seconds.

The use of low temperatures in the primary chamber could affect fuel requirements, the quantity of off-gases, and the use of other kiln technologies.

3.6.1 Test Conditions

The Basin A bench-scale test burns were conducted using a sample from Boring 36-1-3204A because it had the highest concentrations of organics. The following test conditions were employed for the bench-scale burns:

<u>Test</u>	<u>Primary, °C</u>	<u>Afterburner, °C</u>	<u>Residence Time, Sec.</u>	<u>Oxygen, Percent</u>
1	900	1,200	2	7
2	900	1,200	2	5.4
3	800	1,200	2	7
4	800	1,200	2	5.4
5	650	1,200	2	7
6	650	1,200	5	7
7	450	1,200	2	7
8	450	1,200	5	7

A detailed description of the bench-scale test apparatus is provided in the Basin F Laboratory Report and a brief summary in Section 3.1 of this report. A simplified description of the procedure is presented below.

3.6.2 Test Procedure

A 300-gram soil sample was used for all tests. The homogenized sample was placed in the primary chamber and the gas flows regulated to achieve the correct oxygen level for the tests. The secondary burner was brought up to temperature and the system checked. All meters and thermocouples were checked for correct operation. Following this, the temperature controller and rotating mechanism for the primary chamber were turned on. The gases from the primary chamber passed through the fly ash collector, afterburner, and the sampling train. Any fly ash that passed into the sampling train was collected on a prefilter. The following samples were collected for analysis.

Bottom Residue: The bottom residue was made up of all the solid residue from the system including the residue in the primary chamber, the residue in the fly ash separator, and the residue collected in the prefilter to the gas sampling train. This residue was then extracted and analyzed by GC/MS for the target compounds. On selective runs an elemental and trace metal analysis was performed.

Off-Gas: The off-gases were collected on the XAD-2 resin, tenax, and charcoal traps. These traps were combined, digested, and analyzed by GC/MS for target compounds. Metals collected in the various impingers were

analyzed by atomic absorption. The off-gas samples were also analyzed for PICs using the GC/MS-SIM (selective ion mode).

As a result, for each run the feedstock, ash residue, off-gas (target compounds), off-gas (PICs), and metals in the impingers were analyzed. Specifically, the samples were analyzed for organic constituents by GC/MS. For bottom residue and the off gas adsorbents, the POHCs were analyzed by SIM techniques to achieve the low detection limits required. The PICs were determined on the same samples by reanalysis to acquire total ion chromatograms and by performing a computerized library search to identify PICs. The feedstock was analyzed for organic POHCs using total ion chromatogram techniques because the concentration of contaminants in the material was so high and required dilution. The feedstock and metal traps were analyzed for arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver by ICP. Mercury was analyzed by CVAA and the others were analyzed by GFAA.

3.7 EP TOXICITY AND TCLP ANALYSES

The EP Toxicity and TCLP analyses were conducted to quantify the toxicity of the ash residue following incineration. The ash residue was prepared by thermally treating Section 36 samples in a muffle furnace for 1 hour at 900°C (which represents high temperature volatilization) and at 600°C (which represents low temperature volatilization in the primary chamber for the purpose of this research). See Table 3.2-1 for an approximation of time to temperature. The EP Toxicity and TCLP analyses were done on Section 36 samples from Borings 36-1-3239A, 36-17-3086Y, 36-1-3204A, and 36-17-3065A.

3.7.1 EP Toxicity Analysis

The characteristic of EP Toxicity is described in Part 261-Identification and Listing of Hazardous Waste (Section 261.24, Characteristics of EP Toxicity) in 40 CFR 261, Environmental Protection Agency Regulations for Identifying Hazardous Waste, with the specific method described in

Appendix II. The metals and pesticides characterized in the EP Toxicity procedure are presented in Table 3.7-1. The EP Toxicity procedure is briefly outlined below.

A 100-gram sample of thermally treated soil (ash) was obtained from the muffle furnace. The ash was reduced in size as necessary, and extracted with deionized water for 24 hours, while maintaining the pH at or near 5.0. The liquid was then removed for pressurized filtration. The filtered extract was then analyzed for the various metals and pesticides and the results compared to the maximum allowed concentration in Table 3.7-1.

3.7.2 TCLP Analysis

The Toxicity Characteristics Leaching Procedure is a method for determining the mobility of both organic and inorganic contaminants present in liquid, solid, and multiphase water. The various organics and inorganics determined by the TCLP method are divided into the following four categories: pesticides and herbicides, acid extractable phenols, base neutral compounds, and volatile organics. The compounds and the associated regulatory limits are listed by category in Table 3.7-2. The TCLP analysis also includes the EP Toxicity metals as listed in Table 3.7-1. The TCLP analysis was much more complex than the EP Toxicity procedure. The procedure is summarized in Appendix II, Toxicity Characteristic Leaching Procedure in the Federal Register (Vol. 51, No. 114, Friday, June 13, 1980, Proposed Rules, page 21685 to 21693).

TABLE 3.7-1
MAXIMUM CONCENTRATION OF CONTAMINANTS
FOR CHARACTERISTICS OF EP TOXICITY

Contaminant	Maximum Concentration (mg/l)
Metals	
Arsenic	5.0
Barium	100.0
Cadmium	1.0
Chromium	5.0
Lead	5.0
Mercury	0.2
Selenium	1.0
Silver	5.0
Pesticides	
Endrin	0.02
Lindane	0.4
Methoxychlor	10.0
Toxaphene	0.5
2,4-D	10.0
2,4,5-TD, Silvex	1.0

TABLE 3.7-2

MAXIMUM CONCENTRATION OF CONTAMINANTS FOR CHARACTERISTICS
IN THE TCLP ANALYSIS

Compound	Regulatory Limit (mg/l) <u>1/</u>
Pesticides and Herbicides	
Chlordane	0.03
2,4-Dichlorophenoxyacetic acid	1.4
Endrin	0.003
Heptachlor	0.001
Lindane	0.06
Methoxychlor	1.4
Toxaphene	0.07
2,4,5-Trichlorophenoxypropionic acid	0.14
Acid Extractable Phenols	
o-Cresol	10.0
m-Cresol	10.0
p-Cresol	10.0
Pentachlorophenol	3.6
Phenol	14.4
2,3,4,6-Tetrachlorophenol	1.5
2,4,5-Trichlorophenol	5.8
Base Neutrals	
Bis(2-chloroethyl) ether	0.05
1,2-Dichlorobenzene	4.3
1,4-Dichlorobenzene	10.8
2,4-Dinitrotoluene	0.13
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.72
Hexachloroethane	4.3
Isobutanol	36
Nitrobenzene	0.13
Pyridine	5.0
Toluene	14.4

1/ Organic Detection Limits will equal or exceed regulatory limits.

TABLE 3.7-2 (Continued)
MAXIMUM CONCENTRATION OF CONTAMINANTS FOR CHARACTERISTICS
IN THE TCLP ANALYSIS

Compound	Regulatory Limit (mg/l) <u>1/</u>
Volatile Organics	
Acrylonitrile	5.0
Benzene	0.07
Carbon disulfide	14.4
Carbon tetrachloride	0.07
Chlorobenzene	1.4
Chloroform	0.07
1,2-Dichloroethane	0.4
1,1-Dichloroethylene	0.1
Methylene chloride	8.6
Methyl ethyl ketone	7.2
1,1,1,2-Tetrachloroethane	10.0
1,1,1,2-Tetrachloroethane	1.3
Tetrachloroethylene	0.1
1,1,1-Trichloroethane	30.0
1,1,2-Trichloroethane	1.2
Trichloroethylene	0.07
Vinyl chloride	0.05

4.0 RESULTS AND DISCUSSION

This chapter presents and discusses the results of the bench-scale test burns and other research experiments conducted on Section 36 contaminants.

4.1 ORGANIC VOLATILIZATION

As previously discussed, the purpose of the organic volatilization experiment was to determine at what conditions 99.99 percent of the mass of target organics could be volatilized from the soil matrix. The test condition and procedures for this experiment are presented in Section 3.2. The test conditions for the organic volatilization experiments are summarized below:

<u>Run No.</u>	<u>Temperature, °C</u>	<u>Residence Time, min^{1/}</u>
1	200	22.5
2	200	45.0
3	400	15.0
4	400	30.0
5	500	40.0
6	600	7.5
7	600	22.5
8	800	7.5
9	800	15.0

The heatup times are presented in Table 3.2-1 in Section 3.2 of this report. The results of the organic volatilization experiments are presented in Table 4.1-1. This table includes the concentration of target organics in the feedstock as well as the concentration of target organics in the residue after thermal treatment at the various test conditions. The concentration of target organics for Runs Nos. 7, 8, and 9 are not shown because their concentration was below the method detection limits (also shown in Table 4.1-1). The test results for Run Nos. 7, 8, and 9 are presented below.

^{1/} Residence time by definition is the amount of time the soil was at temperature. Residence time does not include the heatup time.

TABLE 4.1-1

CONCENTRATION OF ORGANICS FOR THE ORGANIC VOLATILIZATION
EXPERIMENTS IN THE MUFFLE FURNACE ($\mu\text{g/g}$)^{1,2/}

Compound	Feed- stock ^{3/} $\mu\text{g/g}$	Method Detection Limits $\mu\text{g/g}$	Test Conditions, Temperature/Residence Time					
			200°C/ 22.5 Min $\mu\text{g/g}$	200°C/ 45 Min $\mu\text{g/g}$	400°C/ 15 Min $\mu\text{g/g}$	400°C/ 30 Min $\mu\text{g/g}$	500°C/ 40 Min $\mu\text{g/g}$	600°C/ 7.5 Min $\mu\text{g/g}$
Oxathiane	<65	<0.005	0.04	BDL	0.05	BDL	BDL	BDL
DCPD	<75	<0.005	0.28	0.48	0.24	0.26	BDL	0.06
DIMP	<60	<0.05	BDL ^{4/}	BDL	BDL	BDL	BDL	BDL
DMMP	<150	<0.05	BDL	BDL	1.6	1.1	BDL	0.78
Dithiane	<20	<0.005	BDL	BDL	0.09	0.06	BDL	BDL
DBCP	<20	<0.005	BDL	BDL	BDL	BDL	BDL	BDL
Vapona	<45	<0.05	BDL	BDL	BDL	BDL	BDL	BDL
CPMS	44,600	<0.05	34	44	41	18	BDL	1.4
HCCPD	<20	<0.02	BDL	BDL	BDL	BDL	BDL	BDL
CPMSO	<45	<0.05	9.1	BDL	8.2	6.8	BDL	0.98
CPMSO ₂	<50	<0.05	1.3	2.3	1.6	1.2	BDL	0.57
Atrazine	<220	<0.05	BDL	BDL	BDL	BDL	BDL	BDL
Malathion	<30	<0.05	BDL	BDL	BDL	BDL	BDL	BDL
Aldrin	10,400	<0.03	46	57	43	42	BDL	30
Parathion	<45	<0.05	BDL	BDL	BDL	BDL	BDL	BDL
Isodrin	6,100	<0.01	8.2	13	7.8	7.9	BDL	3.6
Supona	<20	<0.05	BDL	BDL	BDL	BDL	BDL	BDL
DDE	<65	<0.03	BDL	BDL	BDL	BDL	BDL	BDL
Dieldrin	49,000	<0.02	27	44	35	43	BDL	39
Endrin	7,900	<0.05	20	36	16	17	BDL	8.7
DDT	<20	<0.05	BDL	BDL	BDL	BDL	BDL	BDL
Chlordane	<150	<0.01	20	37	15	18	BDL	7.5

^{1/} Sample 36-1-3204A was utilized.

^{2/} All concentrations were calculated on a dry weight basis.

^{3/} The results for the feedstock are at higher detection limits due to dilution.

^{4/} BDL indicates that the concentration in $\mu\text{g/g}$ is at or below the method detection limit for each organic indicated.

<u>Run No.</u>	<u>Temperature, °C</u>	<u>Residence Time, min</u>	<u>Heatup Time, min</u>	<u>Total Time, min</u>
7	600	22.5	26.0	48.5
8	800	7.5	32.0	40.5
9	800	15.0	33.0	48.0

The heatup times for these tests ranged from 26 minutes for the 600°C test condition to 33 minutes for the 800°C test condition. The total time at temperature (above 200°C) was approximately 40 to 50 minutes.

Also of interest was the behavior of the target organics during the low temperature tests (Run Nos. 1 through 6). The percent volatilization of the target organics for compounds present in the feedstock above the method detection limits is presented in Table 4.1-2. As can be seen, the only condition at which a 99.99 percent volatilization was achievable was the run at 500°C with a 40 minute solids residence time. Heatup time to temperature for this test was 23 minutes.

The low temperature runs were not sufficient to volatilize the target compounds, except for the test conditions of 500°C with a residence time of 40 minutes (Run No. 5). It is also clear that lower temperatures were not sufficient to volatilize chlordane, chlorophenylmethlsulfoxide (CPMSO), chorophenylmethysulfone (CPMSO₂), and other organics from the soil matrix (Run Nos. 1, 2, 3, and 4). This also proved true for Run No. 6 at 600° and a residence time of 7.5 minutes.

As a result, the minimum conditions at which low temperature volatilization seems practical are at a temperature of 500°C with a 40-minute solid residence time. A summary of the ability of these test conditions to volatilize target organics is presented in Table 4.1-3. These results are substantiated by the weight loss curve for aldrin shown in Figure 4.1-1. This graph shows that over 90 percent of the aldrin is volatilized at

TABLE 4.1-2
PERCENT VOLATILIZATION OF THE TARGET ORGANICS
PRESENT ABOVE THE DETECTION LIMITS

Compound in Feedstock	Test Conditions, Temperature/Residence Time ^{1/}					
	200°C/ 22.5 min	200°C/ 45 min	400°C/ 15 min	400°C/ 30 min	500°C/ 40 min	600°C/ 7.5 min
CPMS	99.92 ^{2/}	99.90	99.91	99.96	>99.99	>99.99
Aldrin	99.56	99.45	99.59	99.60	>99.99	99.71
Isodrin	99.87	99.79	99.87	99.87	>99.99	99.94
Dieldrin	99.94	99.91	99.93	99.91	>99.99	99.92
Endrin	99.75	99.54	99.80	99.80	>99.99	99.89

^{1/} See Table 3.2-1 for heatup times for various test conditions.

^{2/} The percent of organics volatilized were calculated based on the concentration of target organics present in the feedstock and the thermally treated residue.

TABLE 4.1-3

SUMMARY OF THE ABILITY OF VARIOUS TEST CONDITIONS TO
ADEQUATELY VOLATILIZE TARGET COMPOUNDS

Run No.	Temperature, °C	Residence Time, min	Ability to Volatilize Target Compounds	
			Conditions Sufficient	Conditions Not Sufficient
1	200	22.5		X
2	200	45.0		X
3	400	15.0		X
4	400	30.0		X
5	500	40.0	X	
6	600	7.5		X
7	600	22.5	X	
8	800	7.5	X	
9	800	15.0	X	

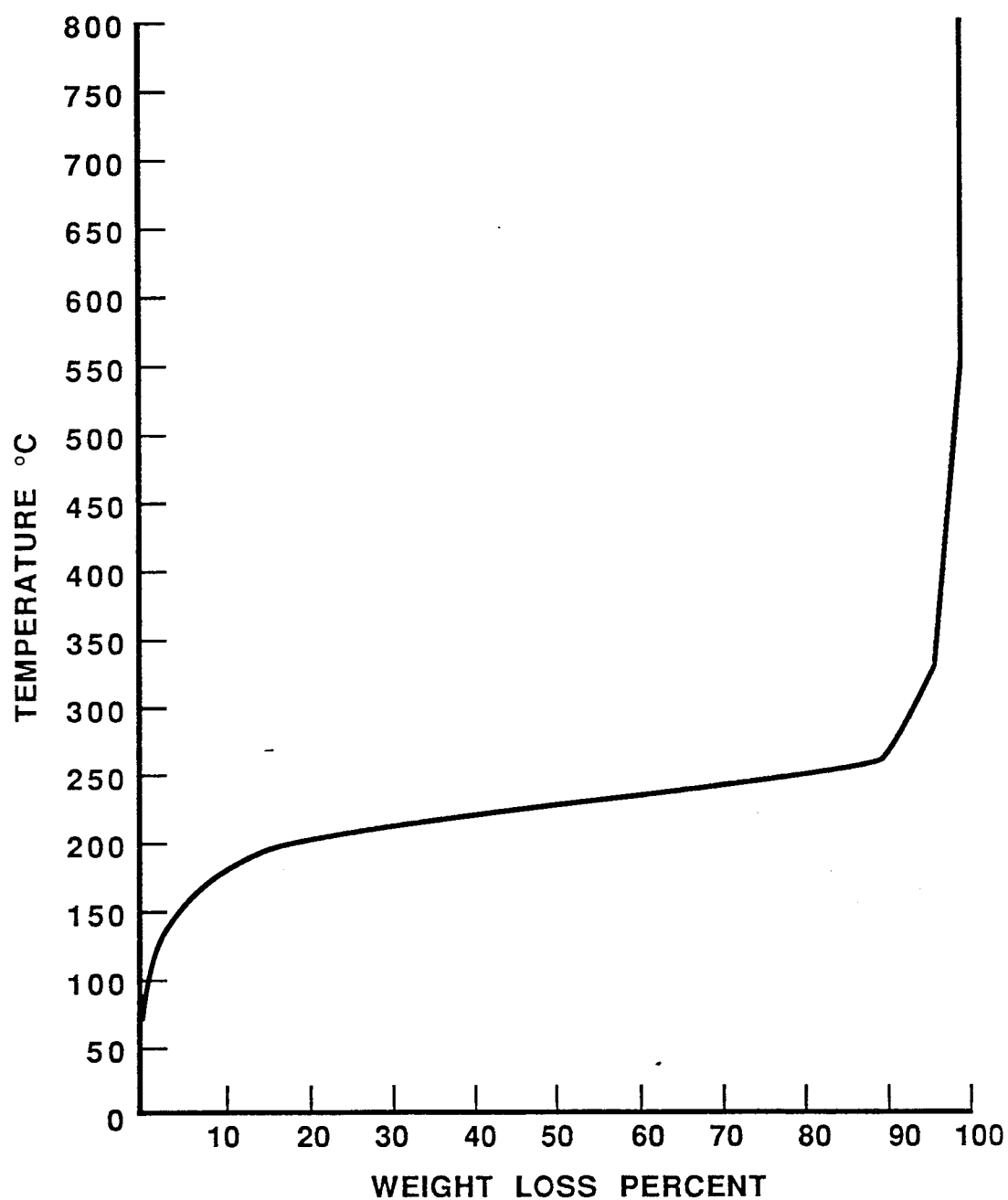


FIGURE 4.1-1
**THERMAL GRAVIMETRIC ANALYSIS OF ALDRIN USING
A HEATUP RATE OF 20°C/MINUTE**

SOURCE: Ferguson et al, 1975

MDMS1261

approximately 250°C at a 15-minute residence time. This graph also indicates a high percent weight loss (greater than 99 percent) around 500°C.

4.2 METAL VOLATILIZATION

The results of the metal volatilization experiments proved inconclusive due to the large variability in the concentration of metals in the feedstock samples from Borings 36-1-3239A and 36-17-3086Y. However, it was possible to determine some trends from the data.

The results indicated that at 400°C over 98 percent of the mercury had volatilized, while at 1,000°C greater than 99.6 percent of the mercury volatilized. These results indicate that mercury will be volatilized to near 100 percent even under low temperature conditions in the primary chamber.

The amount of weight loss between 25°C (ambient conditions) and 200°C varied between 11 and 43 percent on a wet weight basis. The weight loss is mostly associated with the volatilization of moisture.

However, of interest is the cumulative weight loss above 200°C. This is shown below, assuming that the weight loss at 200°C is adjusted to zero (accounting for the removal of moisture). Sample 3204 contained between 6 and 19 percent organics.

	200°C	300°C	400°C	500°C	600°C	800°C
<u>Sample No.</u>	<u>(Percent)</u>	<u>(Percent)</u>	<u>(Percent)</u>	<u>(Percent)</u>	<u>(Percent)</u>	<u>(Percent)</u>
3204	0	N/A	7	N/A	23 ^{1/}	34 ^{1/}
3065	0	0	0	1	1	2
3086	0	5	7	9	10	10

^{1/} Actual salt and metal volatilization between 600 and 800°C was approximately 11 percent on a moisture and organic free basis.

As can be seen, samples 3065 and 3086 have modest weight losses over the range of 200 to 800°C. Sample 3204 has a significant weight loss above 600°C (11 percent), indicating significant volatilization of salts and metals.

4.3 TRAPPING EXPERIMENT

The purpose of the trapping experiment was to determine the rate of volatilization of target organics from the soil matrix. However, because of technical problems (see Section 3.4-1), the primary reactor of the bench-scale unit could not be utilized. Also, Basin A material did not produce sufficient material on the traps, either because of plugging of the primary reactor and/or experimental error in sample selection. As a result, the experiment was conducted in the muffle furnace using Basin F samples. However, the concentration of target organics in the traps was lower than that present in the original feedstock because the muffle furnace could not be adequately sealed.

The amount of target organics collected on the various traps is presented in Table 4.3-1. Due to the loss of volatiles caused by the inability to completely seal the muffle furnace, these are not absolute values and as such they cannot be presented on a total percent basis.

The results indicate that all of the target compounds were volatilized from the soil matrix before the temperature reached 400°C. This can be seen because no organics are present on the trap above the method detection limits. This gives an indication at what temperature the various organics are volatilized. For example, it can be seen that the majority of the chlorophenylmethysulfide (CPMS) and aldrin volatilizes at 200 to 300°C. This can be substantiated for aldrin by information presented in Figure 4.1-1.

4.4 TEMPERATURE PROFILE IN THE BENCH-SCALE UNIT

The results of the temperature profile determination of the primary reactor (of the bench-scale) unit for set point temperatures of 400°C, 650°C, 800°C, and 900°C are presented in Figure 4.4-1. The results indicate that the

3204D

07/11/88

TABLE 4.3-1

THE QUANTITY OF TARGET ORGANICS PRESENT ABOVE THE
METHOD DETECTION LIMITS ON THE TRAPS AT
VARIOUS TEMPERATURES (μg)

Compound	100°C Trap μg	200°C Trap μg	300°C Trap μg	400°C Trap μg
Oxathiane	BDL <u>1/</u>	4.2	0.18	BDL
DCPD	0.54	1.2	BDL	BDL
DBCP	0.08	0.53	BDL	BDL
CPMS	34	42	2.9	BDL
CPMSO	4.5	0.23	BDL	BDL
CPMSO ₂	BDL	0.21	1.3	BDL
Aldrin	0.11	9.11	22	BDL
Isodrin	BDL	0.09	BDL	BDL
Dieldrin	BDL	1.3	0.25	BDL

1/ BDL indicates that the concentration of the particular target compound is less than the method detection limit.

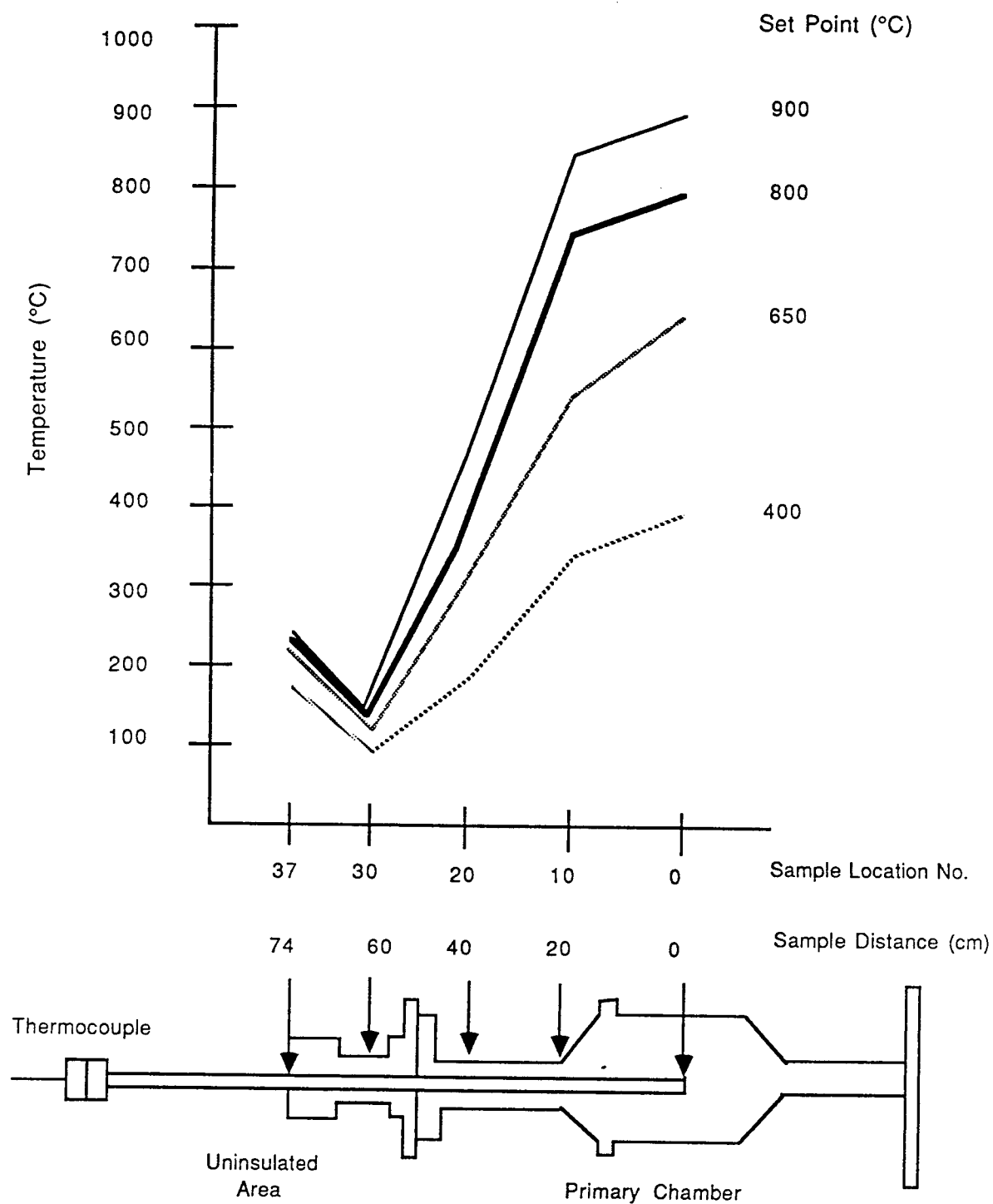


FIGURE 4.4-1

TEMPERATURE PROFILE OF THE PRIMARY REACTOR AS A FUNCTION OF SET POINT TEMPERATURE AND LOCATION ALONG HORIZONTAL AXIS

temperature levels are fairly constant across the primary chamber, but drop fairly rapidly as the thermocouple is withdrawn from the entry to the primary chamber. Condensation reactions could occur in these areas during startup and shutdown, which could lead to the formation of alcohols and ketones as was found for the bench-scale incineration of Basin F soil samples.

4.5 BENCH-SCALE TEST BURNS

Bench-scale laboratory test burns were conducted using the sample from Boring 36-1-3204A because it was found to have the highest concentration of organics. The following conditions were used for the bench-scale test burn.

<u>Test No.</u>	<u>Primary, °C</u>	<u>Afterburner, °C</u>	<u>Residence Time, sec</u>	<u>Oxygen, Percent</u>
1	900	1,200	2	7
2	900	1,200	2	5.4
2	800	1,200	2	7
4	800	1,200	2	5.4
5	650	1,200	2	7
6	650	1,200	5	7
7	450	1,200	2	7
8	450	1,200	5	7

The results of the laboratory bench-scale tests and a discussion of the significance of the results are presented below.

4.5.1 Feed Sample Analysis

A portion of the feedstock sample was analyzed for target organics for each bench-scale test burn. The concentration of target organics present in the feedstock for each bench-scale run is presented in Table 4.5-1. The results indicate the presence of aldrin, isodrin, dieldrin, endrin, and chlordane, as well as chlorophenylmethylsulfide and chlorophenylmethylsulfoxide. All other compounds if present were below the method detection limits of the GC/MS analytical procedure. The sample size used for the test burn was 300 grams. To calculate the weight (in μg) of each compound present, multiply the table value ($\mu\text{g/g}$) by 300 grams.

TABLE 4.5.1

CONCENTRATION OF TARGET ORGANICS IN THE FEEDSTOCK ($\mu\text{g/g}$ or ppm)^{1/}

Test Number	1	2	3	4	5	6	7	8
Primary Temp., °C	900	900	800	800	650	450	650	450
Secondary Temp., °C	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200
Gas Res. Time, sec	2	2	2	2	2	2	5	5
Oxygen, Percent	7	5.4	7	5.4	7	7	7	7
Oxathiane	BDL ^{2/}	BDL	BDL	BDL	BDL	BDL	BDL	BDL
DCPD	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
DIMP	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
DMMP	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Dithiane	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
DBCP	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Vapona	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
CPMS	8,300	BDL	BDL	3,600	9,800	3,200	3,700	4,500
HCCPD	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
CPMSO	BDL	BDL	4,600	BDL	BDL	BDL	BDL	BDL
CPMSO ₂	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Atrazine	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Malathion	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Aldrin	49,000	85,000	55,000	32,000	43,000	50,000	73,000	66,000
Parathion	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Isodrin	3,400	5,300	4,000	6,300	2,400	2,800	4,400	3,900
Supona	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
DDE	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Dieldrin	19,000	76,000	32,000	19,000	18,000	46,000	46,000	36,000
Endrin	4,100	BDL	3,400	3,700	2,600	4,600	7,800	4,300
DDT	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Chlordane	BDL	BDL	6,100	BDL	BDL	BDL	7,700	BDL

^{1/} Sample size was 300 grams of feedstock at 32.7 percent moisture. To calculate the weight of contaminant in total micrograms, multiply the table values above by 300 grams.

^{2/} BDL indicates the compound was present below detection limits.

4.5.2 Residue Analysis

Following thermal treatment, the residues were extracted and analyzed by GC/MS for target organics. Target organics were only detected for a few of the test runs, as indicated in Table 4.5-2.

It is interesting to note that chlorophenylmethylsulfone as well as oxathiane was detected in the ash residue from Run Nos. 5 and 7, even though it was not quantifiable in the feedstock sample analysis. In the Basin F Report, it was pointed out that chlorophenylmethylsulfone and oxathiane were among the more stable contaminants found in the Basin F waste. As such, they could conceivably survive incineration if sufficiently encapsulated in the soil matrix to the point that they experienced depleted oxygen levels or somewhat lower temperatures due to heat transfer limitations (cool regions).

It should also be noted that chlorophenylmethylsulfone is an obvious PIC resulting from the partial oxidation of chlorophenylmethylsulfoxide. This is a likely source of the chlorophenylmethylsulfone because relatively high quantities of the chlorophenylmethylsulfoxide precursor were detected in the soil. Since the chlorophenylmethylsulfoxide would be slowly converted to the chlorophenylmethylsulfone, the resultant chlorophenylmethylsulfone would experience a reduced residence time in the primary chamber and could survive high temperature exposure.

An alternative explanation of the existence of the oxathiane is that it was originally present in the soil, but below detection limits. Because of the high concentration of certain organics, the feedstock required diluting before GC/MS analysis. As a result, the analytical detection limits are higher for the feedstock analysis than the residue analysis, which has a low concentration of organics. As a result, when other organic components of the soil were destroyed, the relatively stable oxathiane survived the high temperatures to be sufficiently above the analytical detection limits for the residue GC/MS analysis.

TABLE 4.5-2

QUANTITY OF TARGET COMPOUNDS PRESENT IN THE ASH RESIDUE (μg)

Test No.	1	5	6	7
Primary Temp., °C	900	650	450	650
Secondary Temp., °C	1,200	1,200	1,200	1,200
Gas Res. Time, sec	2	2	2	5
Oxygen, Percent	7	7	7	7
Aldrin	BDL ^{1/}	BDL	40	10
Dieldrin	6	BDL	BDL	BDL
CPMSO ₂	BDL	10	BDL	18

^{1/} BDL means below detection limits.

4.5.3 Off-Gas Analysis

The off-gas samples were collected on the XAD-2 resin, tenax, and charcoal traps. The material was combined, extracted, and analyzed by GC/MS for target organics. The GC/MS analysis showed that there were no target organics present above the method detection limits. Again, in a few cases very small amounts of chlorophenylmethylsulfone were detected.

4.5.4 Calculation of Destruction and Removal Efficiency

The destruction and removal efficiency of the target compounds was then determined for each of the bench-scale test burns. The DRE was calculated by two methods as indicated below.

The first method of determining the DRE is the method used in the Resource Conservation and Recovery Act (RCRA, Part 264-Standards for Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities, Subpart O - Incinerators). This method is summarized below:

$$DRE = \frac{(W_{IN} - W_{OUT})}{W_{IN}} \times 100$$

where

W_{IN} = Minimum feed rate of target ^{1/} organics in the waste stream feeding the incinerator.

W_{OUT} = Minimum emission rate of the same target organics present in the exhaust emissions prior to release to the atmosphere.

^{1/} Defined as a POHC in RCRA. However, for this work all target compounds were assumed to be POHCs.

Using this method, all of the DREs for the target organics for all tests exceeded 99.9999 percent. This was a result of target organics being found in the off-gases at or below the detection limits. Using this method, the concentration of target compounds present in the ash residue is not considered. However, the ash residue must still pass the EP Toxicity and/or TCLP analysis before it could be disposed of for a full-scale incineration system.

The second calculation method takes into account the concentration of organics present in the ash residue. This method is summarized below:

$$DRE = \frac{(W_{IN} - W_{OUT} - W_{AR})}{W_{IN}} \times 100$$

where

W_{IN} = Minimum feed rate of target organics in the waste feeding the incinerator.

W_{OUT} = Minimum emission rate of the same target organic present in the exhaust emission prior to release to the atmosphere.

W_{AR} = Minimum emission rate of the same target organic present in the ash residue.

The values for W_{AR} can be obtained from Table 4.5-2. As can be seen, the W_{AR} values are small. In fact, the DREs still exceeded 99.9999 percent. The calculated DREs using the above (worst case) method are summarized in Table 4.5-3.

As a result, the DRE exceeded 99.99 percent for all test burns at all conditions tested. This tends to support the idea that lower temperatures in the primary reactor may be possible as long as the residence time is

TABLE 4.5-3

DESTRUCTION AND REMOVAL EFFICIENCY OF TARGET
ORGANICS FOR THE VARIOUS TEST BURNS ^{1/}

Test Number	1	2	3	4	5	6	7	8
Primary Temp., 900 °C	900	900	800	800	650	450	650	450
Secondary Temp., °C	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200
Gas Res. Time, sec	2	2	2	2	2	2	5	5
Oxygen, Percent	7	5.4	7	5.4	7	7	7	7
Oxathiane	BDL ^{2/}	BDL	BDL	BDL	BDL	BDL	BDL	BDL
DCPD	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
DIMP	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
DMMP	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Dithiane	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
DBCP	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Vapona	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
CPMS	>99.99 ^{3/}	BDL	BDL	>99.99	>99.99	>99.99	>99.99	>99.99
HCCPD	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
CPMSO	BDL	BDL	>99.99	BDL	BDL	BDL	BDL	BDL
CPMSO ₂	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Atrazine	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Malathion	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Aldrin	>99.99	>99.99	>99.99	>99.99	>99.99	>99.99	>99.99	>99.99
Parathion	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Isodrin	>99.99	>99.99	>99.99	>99.99	>99.99	>99.99	>99.99	>99.99
Supona	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
DDE	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Dieldrin	>99.99	>99.99	>99.99	>99.99	>99.99	>99.99	>99.99	>99.99
Endrin	>99.99	BDL	>99.99	>99.99	>99.99	>99.99	>99.99	>99.99
DDT	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Chlordane	BDL	BDL	>99.99	BDL	BDL	BDL	BDL	BDL

^{1/} Calculated from the worst case DRE method, which takes into account the concentration of target organics in the bottom residue.

^{2/} BDL indicates the concentration of a specific organic is below the appropriate detection limit.

^{3/} The values are shown to be greater than the 99.99 percent RCRA standard even though all values exceeded 99.9999 percent.

sufficient and as long as the afterburner operates at its optimum condition of 1,200°C gas temperature and a 2 second gas residence time. It appears from these results that a 5-second gas residence time in the afterburner is not necessary.

4.5.5 PIC Analysis

The type and quantity of PICs were analyzed by GC/MS-SIM for the off-gases from each of the bench-scale test burns. The results of the PIC analysis for each of the test burns are presented in Table 4.5-4. Structures of each of the PICs are presented in Appendix B. The total amount of PICs in the off-gases is summarized in Table 4.5-5.

As indicated above, a large number of PICs were identified in this test series at the exhaust of the bench-scale laboratory incineration system. There appears to be no discernible correlation between the observed PICs and the temperature of the primary chamber or the overall oxygen concentration in the exhaust gases. The identity of the specifically observed organic PICs can be grouped into five general categories. The origin of the PICs belonging to these categories is discussed below. A glossary of chemical nomenclature used in the following discussion is presented in Appendix C.

Substituted Phthalates

Various phthalic acid esters (e.g., diisooctyl phthalate) were observed. Reaction kinetic data have been previously obtained on a number of phthalates, and it has been demonstrated that their decomposition may proceed by a low energy five-center elimination reaction (Benson and O'Neal 1970). Thus, it is expected that these compounds will be destroyed at temperatures of about 600°C and residence times of 2.0 seconds. It seems highly unlikely that the observed phthalates could be combustion-related PICs.

TABLE 4.5-4
CONCENTRATION OF PICS
FOR THE VARIOUS TEST BURNS (ug)

Test Number	1	2	3	4	5	6	7	8
Primary Temp., °C	900	900	800	800	650	450	650	450
Secondary Temp., °C	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200
Gas Res. Time, sec	2	2	2	2	2	2	5	5
Oxygen, Percent	7	5.4	7	5.4	7	7	7	7
Bis (2 methoxyethyl 1) Phthalate	0.6	1.8	BDL	BDL	BDL	BDL	BDL	BDL
Butyl-2-methylpropyl Phthalate	2.1	BDL	BDL	BDL	BDL	BDL	BDL	BDL
5-Butyl-Nonane	BDL ^{1/}	BDL	BDL	BDL	0.8	BDL	BDL	BDL
2,5-Cyclohexadiene-1,4-dione	0.5	BDL	BDL	BDL	BDL	BDL	0.5	BDL
Decamethyl-Cyclopentasiloxane	1.2	1.0	BDL	0.7	BDL	BDL	1.3	BDL
Decane	0.5	1.3	BDL	BDL	BDL	BDL	BDL	BDL
Diisooctyl Phthalate	1.0	BDL	BDL	BDL	BDL	0.4	BDL	BDL
2,2-Dimethyl-Decane	BDL	BDL	BDL	BDL	3.2	BDL	1.1	BDL
3,6-Dimethyl-Decane	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.6
2,2-Dimethyl-Heptane	BDL	1.1	BDL	BDL	BDL	0.9	BDL	4.9
3,7-Dimethyl-Heptane	BDL	BDL	BDL	BDL	BDL	0.8	BDL	BDL
3,7-Dimethyl-Nonane	0.4	BDL	BDL	BDL	BDL	BDL	BDL	BDL
2,8-Dimethyl-Undecane	BDL	BDL	BDL	BDL	BDL	BDL	BDL	1.0
3,6-Dimethyl-Undecane	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.8
3,8-Dimethyl-Undecane	BDL	1.3	BDL	BDL	BDL	BDL	BDL	BDL
Dodecamethyl-Cyclohexasiloxane	0.6	0.5	BDL	BDL	BDL	BDL	0.7	BDL
Eicosane	1.7	BDL	BDL	BDL	BDL	BDL	BDL	BDL
6-Ethyl-2-Methyl-Octane	0.9	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Heptadecane	BDL	BDL	BDL	0.5	BDL	BDL	BDL	BDL
Hexadecanol (Cetyl Alcohol)	BDL	BDL	BDL	0.5	BDL	BDL	BDL	BDL
Hexamethyl-Heptane	BDL	1.5	BDL	BDL	BDL	BDL	1.6	0.7
Hexanol	BDL	BDL	BDL	BDL	0.5	BDL	BDL	BDL
Hexanedioic Acid	0.7	BDL	BDL	BDL	BDL	BDL	0.6	BDL
10-Methyl-Eicosane	BDL	BDL	BDL	BDL	3.4	BDL	BDL	BDL
3-Methyl-5-Propyl-Nonane	BDL	BDL	BDL	BDL	BDL	BDL	0.9	4.1
12-Methyl-Tridecanol	BDL	0.5	BDL	BDL	BDL	BDL	BDL	BDL
Nonadecane	BDL	BDL	BDL	0.5	BDL	BDL	BDL	BDL
Octadecen-1-ol	BDL	BDL	BDL	BDL	BDL	0.9	0.6	4.2
Octamethyl-Cyclotetrasiloxane	BDL	1.7	BDL	1.3	2.5	2.4	BDL	BDL
Phenol	1.7	BDL	BDL	0.6	BDL	BDL	BDL	BDL
1,13-Tetradecadiene	1.9	3.0	1.9	BDL	BDL	BDL	BDL	BDL
Tetramethyl-Heptane	BDL	0.4	BDL	BDL	BDL	BDL	BDL	BDL
Tetramethyl-Hexane	BDL	BDL	BDL	BDL	4.0	BDL	BDL	BDL
2,2,5,5-Tetramethyl-Hexane	2.0	BDL	BDL	BDL	BDL	BDL	0.9	BDL
Tetramethyl-Pentane	BDL	BDL	BDL	BDL	0.5	BDL	BDL	BDL
2,2,4-Trimethyl-Decane	0.5	BDL	BDL	BDL	BDL	BDL	BDL	1.3
2,2,7-Trimethyl-Decane	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.4
2,2,9-Trimethyl-Decane	1.6	BDL	BDL	BDL	BDL	BDL	BDL	3.7
2,5,9-Trimethyl-Decane	0.5	BDL	BDL	BDL	BDL	BDL	BDL	BDL
2,6,8-Trimethyl-Decane	BDL	BDL	BDL	BDL	0.6	BDL	BDL	BDL
2,2,4-Trimethyl-Heptane	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.5
2,3,4-Trimethyl-Heptane	BDL	BDL	BDL	BDL	BDL	BDL	BDL	3.1
2,5,5-Trimethyl-Heptane	BDL	BDL	BDL	BDL	1.8	BDL	BDL	BDL
2,2,4-Trimethyl-Hexane	1.4	BDL	BDL	BDL	0.8	0.9	BDL	BDL
2,2,5-Trimethyl-Hexane	BDL	BDL	BDL	BDL	BDL	0.6	BDL	BDL
2,3,4-Trimethyl-Hexane	BDL	0.7	BDL	BDL	BDL	BDL	BDL	BDL
2,2,6-Trimethyl-Octane	0.4	BDL	BDL	BDL	0.5	BDL	BDL	0.5
2,3,6-Trimethyl-Octane	BDL	BDL	BDL	BDL	BDL	0.5	BDL	BDL
Undecane	0.6	0.4	BDL	BDL	0.9	BDL	BDL	0.8

1/ BDL means below detection limits.

3204D

TABLE 4.5-5
TOTAL PICS IDENTIFIED (μg)

Test Number	1	2	3	4	5	6	7	8
Primary Temp., °C	900	900	800	800	650	450	650	450
Secondary Temp., °C	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200
Gas Res. Time, sec	2	2	2	2	2	2	5	5
Oxygen, Percent	7	5.4	7	5.4	7	7	7	7
Sulfur, Mol. (μg)	3.5	0.7	BDL ^{1/}	BDL	BDL	BDL	1.3	BDL
Identified PICS (μg)	22.0	15.2	1.9	4.1	20.1	7.4	8.2	26.6
Unknown PIC (μg)	<u>4.6</u>	<u>5.1</u>	<u>1.3</u>	<u>0.9</u>	<u>5.0</u>	<u>0.8</u>	<u>4.8</u>	<u>1.2</u>
Total PICS (μg)	30.1	21.0	3.2	5.0	25.1	8.2	14.3	27.8

^{1/} BDL means below detection limits.

In fact, researchers at University of Dayton Research Institute (UDRI) have observed that phthalates may be produced as artifacts in GC analyses. The septum material commonly used in GC inlet ports contain phthalates. Pieces of septum material are sometimes dislodged and heated in the inlet ports of the GC, which can result in volatilization of the phthalates or decomposition of the phthalate-containing material in the septum. It is also possible that phthalates may be introduced as impurities from other laboratory sources or be present in the ambient air, as they are common components in most plasticizing materials. This same information has been used to explain the observation of phthalates in the effluent of hazardous waste incinerators (Trenholm and Hathaway 1984).

Siloxanes

Several siloxane isomers were also observed from the bench-scale incineration tests. These have also been observed by other researchers, including those at UDRI. The observation of these siloxanes has been attributed to degradation of GC column material by the corrosive materials being analyzed. Siloxanes have been commonly observed when no silicon-containing precursors are known to be present. Considerable experience is available in this area, and it appears quite certain that the observations of siloxanes is due to degradation of quartz tubing in the GC columns and afterburner of the bench-scale unit. Siloxanes should not be considered a true PIC.

Substituted Alkanes and Alkenes

The vast majority of the observed PICs were alkanes (e.g., undecane) and alkenes (1,13-tetradecadiene). The origin of these compounds is of little practical importance because they are not considered toxic. Furthermore, they are very unlikely to be formed as PICs from the combustion process. Such branched-chain hydrocarbons have many reactive hydrogens present and relatively weak carbon-carbon bonds, which result in their being relatively fragile compounds. It is conceivable that they are formed by recombination

reactions of radical fragments in the cooler transport line downstream of the primary and secondary chambers. However, our current understanding of kinetics suggests that a sequence of reactions resulting in formation of such large molecules is improbable.

However, it is puzzling that these molecules were not observed in the previous laboratory studies. This suggest a possible experimental artifact or an unknown source of contamination. PICs such as this have not been previously observed in any tests conducted at UDRI.

Partially Oxidized Alkanes

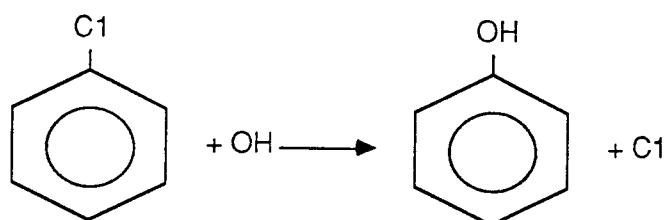
Research studies have shown that the primary products formed in the oxidation of organic compounds for temperatures up to 400°C are alcohol, carbonyls, and carboxylic acids. Compounds such as 2,5-cyclohexadiene-1,4,dione; hexadecanal; hexanal/hexanedioic acid; 12-methyl-tridecanal; and octadecen-1-ol are products that may be obtained by the partial oxidation of the corresponding hydrocarbon. However, at temperatures around 400°C, these reactions proceed through attack by molecular oxygen or organic peroxides, and residence times on the order of minutes may be required to achieve observable yields of products. This would mean that the parent alkane would have to be held up in the transport lines of the system for a few minutes for the observed products to be formed. This is entirely possible for this type of system, which requires collection of the effluent over an extended period and for which quantitative transport is difficult to achieve due to possible "cold spots" in the transfer line.

Consequently, it is believed that these partially oxidized hydrocarbons are being produced in the transport lines of the combustor at relatively low temperatures by reaction with molecular oxygen and initially formed peroxides.

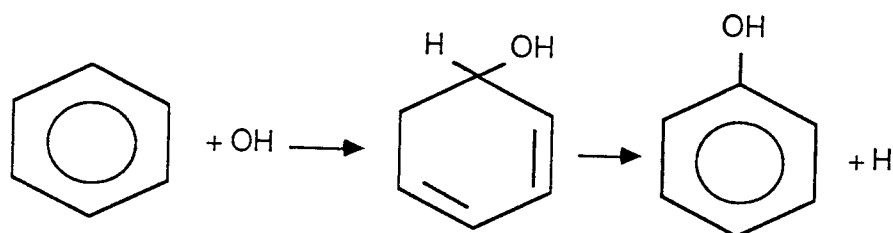
Phenol

Phenol is the sole observed PIC that may be attributed to formation in the combustion zones of the system. An aromatic precursor such as benzene is too stable to be oxidized by the mechanisms discussed in the previous paragraphs. Instead, hydroxyl radical (OH), which is formed at temperatures above 600°C, is required for rapid oxidation of benzene to form phenol. Any of the contaminants (or naturally occurring organics) in the soil could furnish the necessary phenyl-containing precursor for the formation of phenol.

Phenol is a commonly observed combustion product in field and laboratory combustion of toxic organics (Trenholm and Hathaway 1984; Graham et al. 1986). It may be formed by displacement reactions by OH on substituted benzenes (e.g., toluene, chlorobenzene, etc.) or direct addition to benzene with subsequent loss of a hydrogen atom (see Reactions 1 and 2).

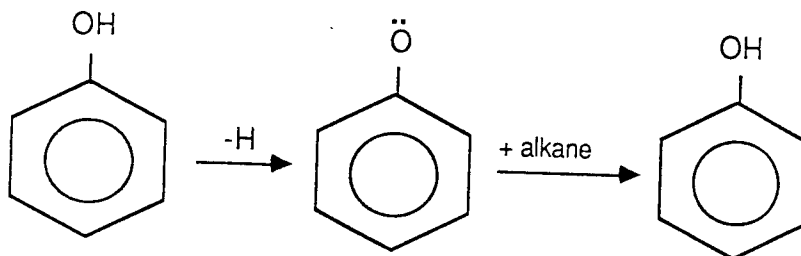


Reaction 1



Reaction 2

Once formed, the phenol molecule is expected to lose the phenolic hydrogen rather easily (bond dissociation energy of 86.5 kcal/mole). However, the phenolic radical is quite stable (heat of formation of 11.4 kcal/mole) and may survive the combustion zones until it can abstract a hydrogen from another organic in the cooler transport zones and reform phenol (see Reaction 3) (McMillen and Golden 1982).



Reaction 3

Dioxins and Furans

The off-gas samples from several of the bench scale test runs (Run Nos. 1, 3, 5, and 7) were analyzed for polychlorinated dibenzo-dioxin (PCDD) and polychlorinated dibenzo-furans (PCDF). The concentrations of these compounds were below the detection limits (1 ppb) in all the test burns except for Run No. 1, where polychlorinated dibenzo-dioxin was present at a concentration of 1.4 ppb. The concentrations of the isomers of dioxins and furans present in the off-gases need to be fully evaluated during the pilot plant phase.

4.5.6 Metal Analysis

Metal analyses were performed on the material collected in the impingers of the gas cooling train. The results of the metal analysis proved inconclusive because of the occurrence of a reverse matrix suppression effect for arsenic, chromium, lead, selenium, and silver. Also, analytical spike recoveries were below 40 percent in each case analyzed.

As a result, a second experiment was performed to overcome this problem. For this analysis, feedstock from sample 3204 was analyzed for ICP metals. Following this, samples were ashed at 600°C for 1 hour and at 900°C for 1 hour. The ash residue was then similarly analyzed for ICP metals. The results of the analysis are presented in Table 4.5-6. The concentrations in this table were adjusted to take into account moisture in the feedstock and also the loss of weight of the ash during the ashing period; placing the results on a common basis. The results seem to indicate that arsenic, mercury, and sodium are volatilized during the ashing period to a relatively large degree, whereas cobalt and copper are volatilized to only a slight degree. It is difficult to discern the volatilization of other metals because of the large amount of variation in the feedstock.

The results appear reasonable since arsenic sublimates at 615°C, while mercury vaporizes at 357°C and sodium at 890°C. As can be seen, there is a distinct drop in the concentration of sodium between 600°C and 900°C, while the mercury concentration stays approximately the same. Also, cobalt and copper have significantly higher boiling points and have not been shown to highly partition in some cases.

These results should be viewed with caution since ashing in the muffle furnace does not represent the following mechanisms associated with a flame mode incinerator:

- 1) Vaporization and subsequent condensation reactions of metals in cooler regions of an incinerator:
- 2) Eutectic effects; and
- 3) The effects of oxygen levels at combustion conditions.

4.6 EP TOXICITY AND TCLP ANALYSES

The EP Toxicity and TCLP analyses were conducted on thermally treated residues prepared in a muffle furnace by ashing the material at 900°C for

TABLE 4.5-6
CONCENTRATION OF ICP METALS IN FEEDSTOCK
AND THE THERMALLY TREATED RESIDUE
($\mu\text{g/g}$)

Metal	Feedstock ^{1/}	Concentration in Solid Residue	
		600°C for 1 Hour ^{2/}	900° for 1 Hour ^{3/}
Aluminum	5,510-6,100	7,055	9,590
Antimony	<9.0	<3.5	3.3
Arsenic	144-313	148	153
Barium	70-92	77	99
Beryllium	<1.8	1.0	1.0
Cadmium	<3.7	<1.5	<1.4
Calcium	259,000-275,000	287,000	329,000
Chromium	10-12	9.1	12
Cobalt	<7.7-13	3.0	<2.8
Copper	16-22	14.9	13
Iron	3,870-4,910	4,220	4,570
Lead	15	12	10
Magnesium	1,690-1,950	1,870	2,080
Manganese	72-97	82	100
Mercury	1.4-1.6	<0.1	0.1
Nickel	21-43	24	28
Potassium	544-596	860	1,100
Selenium	<4.5	<2.0	1.7
Silver	<9.1	<4.0	3.4
Sodium	2,100-2,550	1,720	1,460
Thallium	<9.1	3.2	3.3
Vanadium	50-67	48	66
Zinc	27-32	34	25

^{1/} Feedstock values are on a dry weight basis.

^{2/} Concentration adjusted for weight loss during ashing of 17 percent.

^{3/} Concentration adjusted for weight loss during ashing of 30 percent.

60 minutes. Samples from the bench-scale tests could not be used since this material was extracted and analyzed by GC/MS for target organics.

The results of the metal analysis portion of the EP Toxicity test compared to the regulatory limits are presented in Table 4.6-1. In all cases the concentration of metals was well below the regulatory limit. The results from the TCLP analysis were essentially identical.

Pesticides were also analyzed for by the EP Toxicity and TCLP methods. Pesticides tested for included chlordane, endrin, heptachlor, lindane, methoxychloro, and toxaphene. The analytical results, appropriate method detection limits, and maximum allowed concentration for both the EP Toxicity and TCLP analysis are presented in Table 4.6-2.

The analytical results indicate that no pesticides were present down to the detection limits for both the EP Toxicity and TCLP analyses for the thermally treated samples.

In conclusion, the concentrations of metals in the EP Toxicity and TCLP tests were a factor of 10 below the maximum regulated concentrations. The concentrations of regulated pesticides were below the method detection limits and, as a result, significantly below the maximum regulated concentrations for both the TCLP and EP Toxicity analyses.

TABLE 4.6-1

ANALYTICAL RESULTS OF THE EP TOXICITY ANALYSIS
ON SECTION 36 SOIL SAMPLES (ppb)

Sample ^{1/}	Arsenic	Barium	Cadmium	Chromium	Lead	Mercury	Selenium	Silver
3065	<500	<10,000	<100	<500	<500	<20	<100	<500
3086	<500	<10,000	<100	<500	1,390	<20	<100	<500
3204	<500	<10,000	<100	<500	<500	<20	<100	<500
3239	530	<10,000	<100	<500	<500	<20	<100	<500
3204 ^{2/}	<500	<10,000	<100	<500	<500	<20	<100	<500
3204 ^{2/}	<500	<10,000	<100	<500	<500	<20	<100	<500
Maximum Limit ^{3/}	5,000	100,000	1,000	5,000	5,000	200	1,000	5,000

^{1/} Sample numbers compare to boring numbers in Chapter 2.0.

^{2/} Sample prepared by ashing 600°C for 40 minutes.

^{3/} This represents the maximum concentration limit for both the EP Toxicity and TCLP analyses.

TABLE 4.6-2

METHOD DETECTION LIMITS AND MAXIMUM ALLOWED REGULATED
CONCENTRATIONS OF PESTICIDES FOR THE EP TOXICITY AND TCLP ANALYSES

Compound	EP Toxicity Analysis		TCLP Analysis	
	Detection Limit (ppb) <u>1/</u>	Maximum Regulated Concentration (ppb) <u>2/</u>	Detection Limit (ppb) <u>1/</u>	Maximum Regulated Concentration (ppb) <u>2/</u>
Chlordane	N/A <u>3/</u>	N/A	<20.5	30
Endrin	<0.2	20	<0.1	3
Heptachlor	N/A	N/A	<0.1	1
Lindane	<0.1	400	<0.05	60
Methoxychloro	<0.1	10,000	<0.5	1,400
Toxaphene	<2.0	500	<1.0	70

1/ Detection limit of the GC/MS procedure.

2/ Maximum concentrations allowed for the EP Toxicity and TCLP analyses.
Concentrations above these values fail the appropriate test.

3/ Not applicable to EP Toxicity analysis.

5.0 SUMMARY AND CONCLUSIONS

As discussed in the introduction of this report, the objectives of this laboratory program were to determine the following: 1) the degree of volatilization of organics from the soil matrix; 2) the degree of metal partitioning occurring; 3) the DRE of target organics and PICs for test burns in the bench-scale laboratory unit; and 4) the ability of the thermally treated residue to pass the EP Toxicity and TCLP analyses.

Experiments on organic volatilization of target compounds from the soil matrix were conducted in the muffle furnace (to model the primary chamber of a rotary kiln). The muffle furnace experiments indicated that in excess of 99.99 percent of the target organics could be volatilized at 500°C with a 40-minute residence time. The results indicated that lower temperatures were insufficient to volatilize chlordane, chlorophenylmethylsulfide, chlorophenylmethylsulfone, aldrin, and other organics from the soil matrix. The inability to volatilize pesticides to a sufficient degree at lower temperatures has also been shown to be true by other researchers (Ferguson et al. 1975), an example of which was presented for aldrin. The trapping experiment also gave similar results. This test indicated that significant amounts of chlorophenylmethylsulfide, chlorophenylmethylsulfone, and aldrin were still leaving the soil matrix at 300°C. In fact, the majority of aldrin appears to leave at 300°C.

The results of the metal volatilization tests proved inconclusive because of large variability in sample composition. However, the results indicated that in excess of 90 percent of the mercury leaves the soil matrix at 400°C and 99.6 percent by 800°C. Incinerators operated at relatively low temperatures would volatilize a significant amount of the mercury. The results also indicated a significant variation in weight loss of the thermally treated residues. Weight loss on a moisture and organic free basis of the residue ranged from a low of 2 percent to a high of 11 percent depending on boring sample.

All of the bench-scale test burns indicated that in excess of 99.9999 percent of the target organics were destroyed. An insignificant amount of target organics were detected in the residue and the concentration of target organics was below the method detection limits for the off-gases. Most of the PICs identified were not combustion related, but were related to the method of analysis and possible low temperature condensation reactions. The only combustion related PIC observed was phenol in the high temperature runs of 800°C and 900°C in the primary chamber. The results of the metal analysis from the bench-scale runs indicated that arsenic, mercury, and sodium are readily volatilized from the soil matrix.

The EP Toxicity and TCLP analyses were conducted on the thermally treated material. The results indicated that the thermally treated residue would pass the EP Toxicity and TCLP analyses for metals and pesticides.

In conclusion, the results of the subject research indicate the following points:

- 1) The target organics can be readily volatilized from the soil matrix at temperatures as low as 500°C. As a result, further investigations into low temperature volatilization are desirable.
- 2) The metal analyses conducted indicate that the concentration of metals in the residues is highly variable, as is the weight loss at high temperatures.
- 3) The target compounds were readily destroyed in the bench-scale unit for all temperature regimes with a minimal generation of combustion-related PICs.
- 4) The 900°C temperature in the primary reactor and a 2-second gas residence time in the afterburner at 1,200°C seems appropriate to achieve a 99.99 DRE as it was for Basin F material.

- 5) The thermally treated residue passed the EP Toxicity and TCLP analyses.

Responses to comments from Shell Oil Company and EPA Region VIII are presented in Appendix D.

Appendix A

Chemical Structures To 22 Semivolatile
Organic Target Compounds

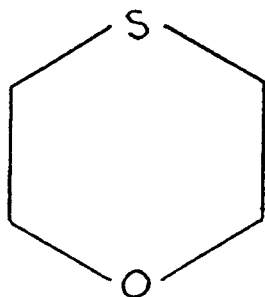
CROSS REFERENCE TO STRUCTURES OF COMPOUNDS TESTED FOR
IN THE BENCH-SCALE LABORATORY TEST PROGRAM

1. Oxathiane - Thioxane, C_4H_8SO , 1,4-Oxathiane
2. DCPD - dicyclopentadiene, $C_{10}H_{12}$, 3a,4,7,7a-Tetrahydro-4,7-methanoindene
3. DIMP - diisopropylmethylphosphonate, $C_7H_{17}O_3P$
4. DMMP - dimethylmethylphosphonate, $C_3H_9O_3P$
5. Dithiane - Nabam, $C_4H_6N_2Na_2S_4$, Ethylenebis(dithiocarbamic acid)disodium salt
6. DBCP - Nemagon, dibromochloropropane, $C_3H_5Br_2Cl$, 3-Chloro-1,2-dibromopropane
7. Vapona - dichlorvos, $C_4H_7Cl_2PO_4$ 0,0-dimethyl 0-(2,2-dichlorovinyl phosphate)
8. PCPMS - p-chlorophenylmethylsulfide, C_7H_7ClS
9. HCCPD - C_5Cl_6 , hexachlorocyclopentadiene
10. PCPMSO - p-chlorophenylmethylsulfoxide, C_7H_7ClSO
11. PCPMSO₂ - p-chlorophenylmethylsulfone, $C_7H_7ClSO_2$
12. Atrazine - $C_8H_{14}N_5Cl$, 2-chloro-4-ethylamino-6-isopropylamino-s-triazine
13. Malathion - $C_{10}H_{19}O_6PS_2$, S - (1-2 dicarbethoxyethyl)0,0-dimethyldithiophosphate
14. Aldrin - $C_{12}H_8Cl_6$, 1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5,8-dimethanonaphthalene
15. Parathion - $C_{10}H_{14}NO_5PS$, 0,0-diethyl 0-p-nitrophenyl phosphorothioate
16. Isodrin - $C_{12}H_8Cl_6$, 1,2,3,4,10,10-hexachloro-1,4,4a,8,8a-hexahydro-1,4:5,8-endo-dimethanonaphthalene
17. Supona - Chlorfenvinphos, $C_{12}H_{14}Cl_3O_3P$, 0,0-diethyl 0-[2-chloro-1-(2,4-dichlorophenyl)vinyl] phosphate
18. P,P'-DDE - $C_{14}H_8Cl_4$, 1,1-dichloro-2,2-bis-(p-chlorophenyl)ethylene

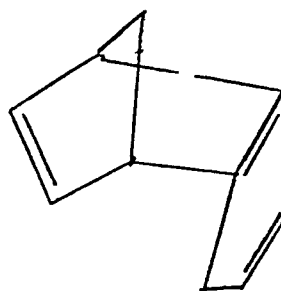
CROSS REFERENCE TO STRUCTURES OF COMPOUNDS TESTED FOR
IN THE BENCH SCALE LABORATORY TEST PROGRAM (Continued)

19. Dieldrin - $C_{12}H_8Cl_6O$, 1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4,5,8-dimethanonaphthalene
20. Endrin - $C_{12}H_8Cl_6O$, 1,2,3,4,10,10-hexachloro-6,7-epoxy, 1,4,4a,5,6,7,8,8a-octahydro-endo-endo-1,4:5,8-dimethanonaphthalene
21. P,P'-DDT - dichloro diphenyl trichloroethane $(C_6H_4)_2CHCCl_3$, 1,1,1-trichloro-2,2-bis(p-chlorophenyl)ethane
22. Chlordane - $C_{10}H_6Cl_8$, 1,2,4,5,6,7,8,8-octachloro-4,7,7a-methane-3a,4,7,7a-tetrahydroindane

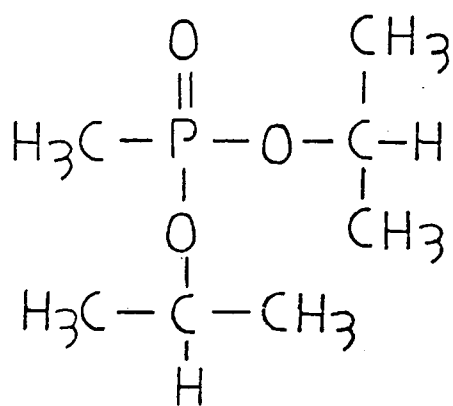
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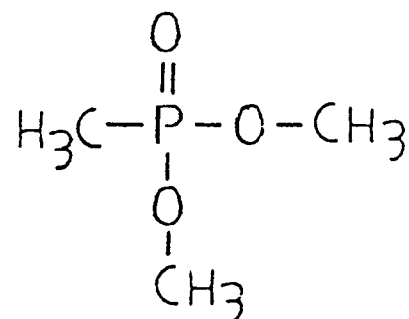
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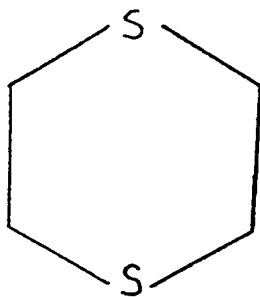
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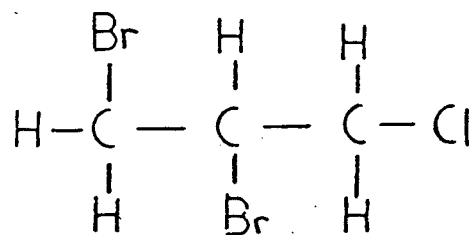
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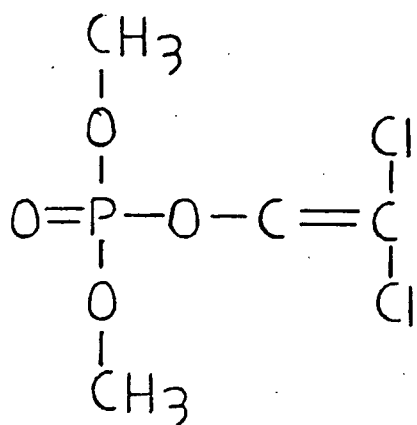
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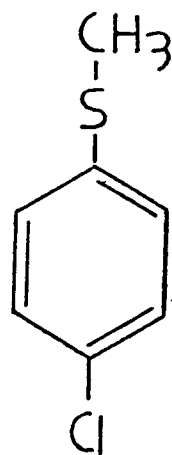
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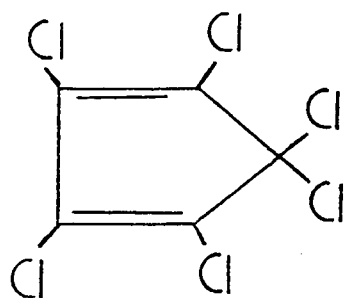
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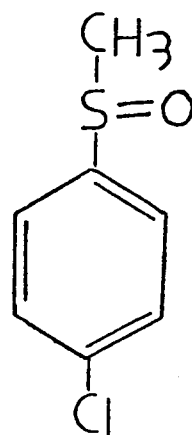
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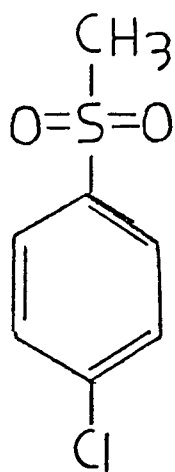
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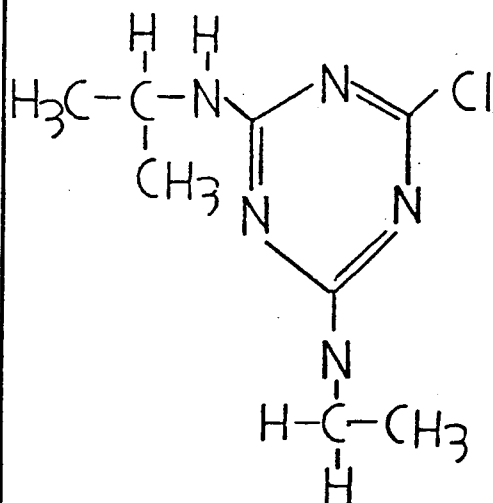
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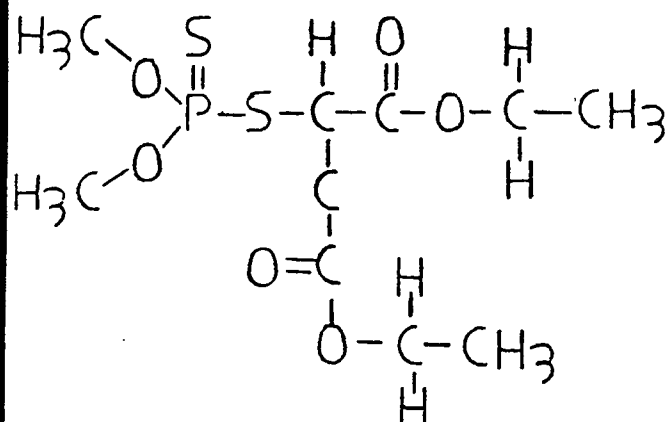
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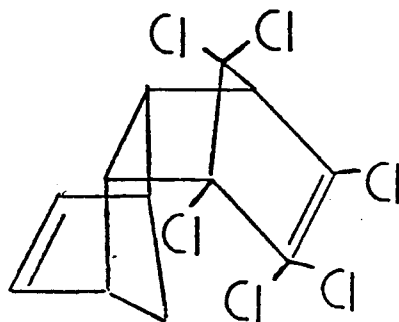
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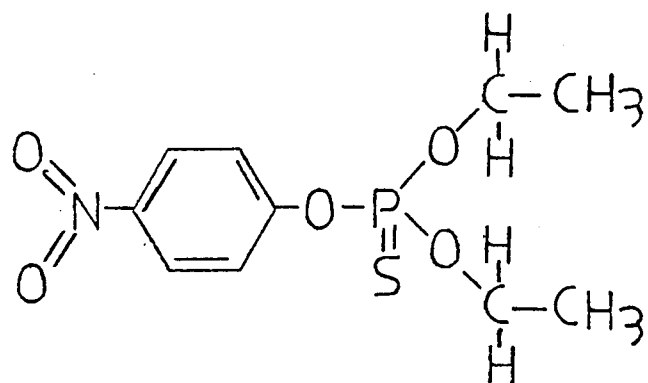
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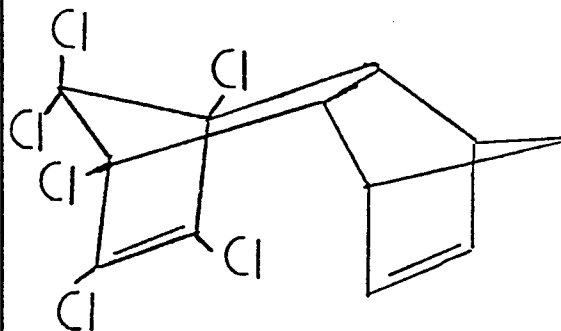
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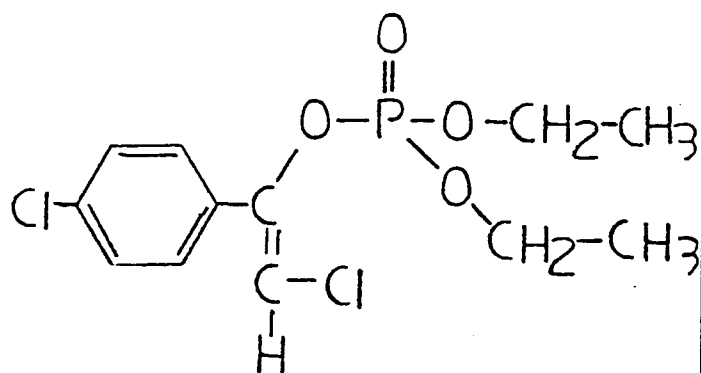
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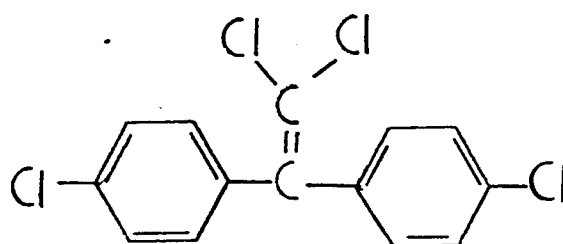
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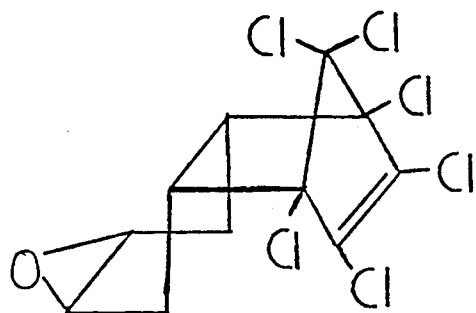
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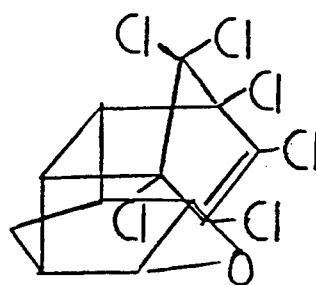
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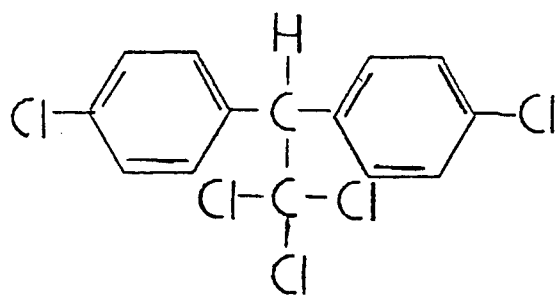
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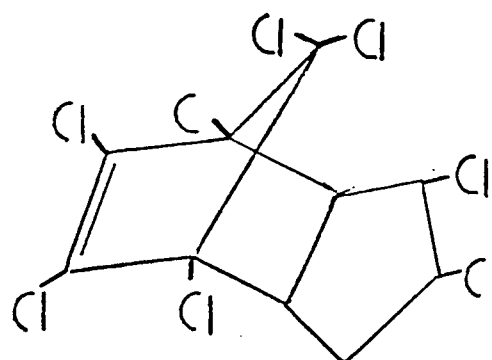
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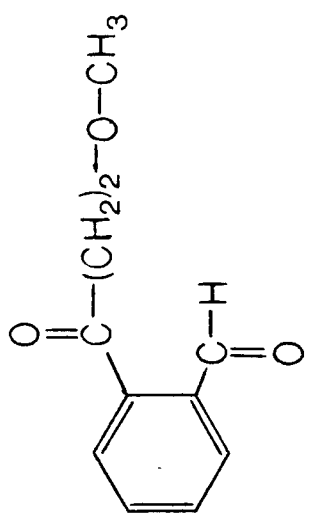
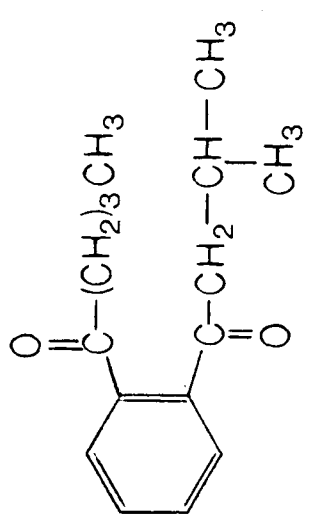
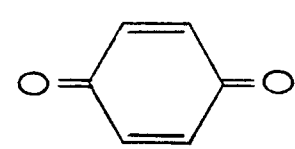
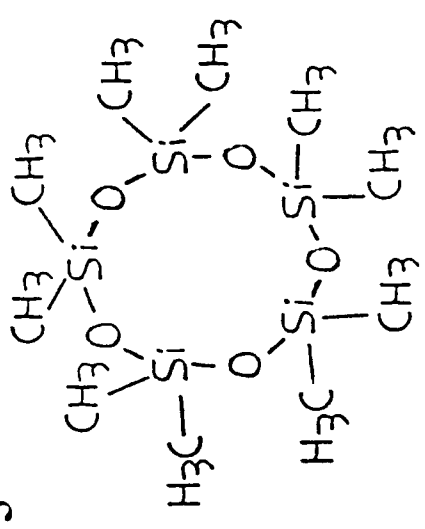
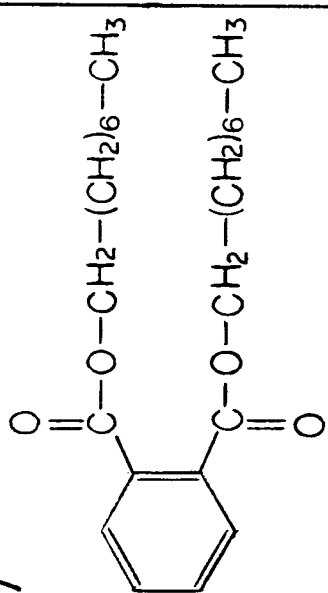
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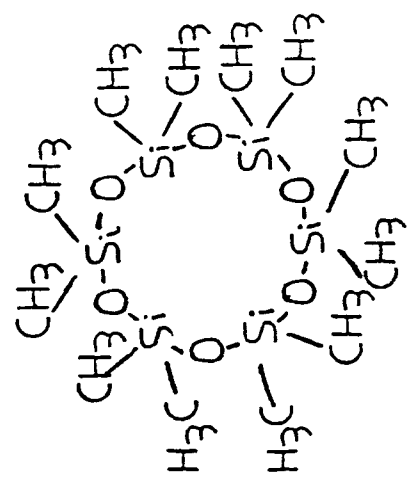
Appendix B

Alphabetized Cross Reference To The Structures Of Products Of Incomplete Combustion

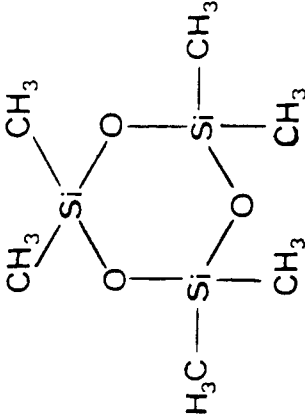
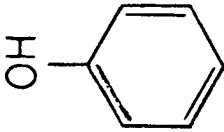
ALPHABETIZED CROSS REFERENCE TO THE STRUCTURES OF PRODUCTS OF INCOMPLETE COMBUSTION (PICS)

1.	Bis (2 methoxyethyl) Phthalate	26.	12-Methyl-Tridecano1
2.	Butyl-2-methylpropyl Phthalate	27.	Nonadecane
3.	5-Butyl-Nonane	28.	Octadecen-1-ol
4.	2,5-Cyclohexadiene-1,4-dione	29.	Octamethyl-Cyclotetrasiloxane
5.	Decamethyl-Cyclopentasiloxane	30.	Pheno1
6.	Decane	31.	1,13-Tetradecadiene
7.	Diisooctyl Phthalate	32.	Tetramethyl-Heptane
8.	2,2-Dimethyl-Decane	33.	Tetramethyl-Hexane
9.	3,6-Dimethyl Decane	34.	2,2,5,5-Tetramethyl-Hexane
10.	2,2-Dimethyl-Heptane	35.	Tetramethyl-Pentane
11.	3,7-Dimethyl-Heptane	36.	2,2,4-Trimethyl-Decane
12.	3,7-Dimethyl-Nonane	37.	2,2,7-Trimethyl-Decane
13.	2,8-Dimethyl-Undecane	38.	2,2,9-Trimethyl-Decane
14.	3,6-Dimethyl-Undecane	39.	2,5,9-Trimethyl-Decane
15.	3,8-Dimethyl-Undecane	40.	2,6,8-Trimethyl-Decane
16.	Dodecamethyl-Cyclohexasiloxane	41.	2,2,4-Trimethyl-Heptane
17.	Eicosane	42.	2,3,4-Trimethyl-Heptane
18.	6-Ethyl-2-Methyl-Octane	43.	2,5,5-Trimethyl-Heptane
19.	Heptadecane	44.	2,2,4-Trimethyl-Hexane
20.	Hexadecano1 (Cetyl Alcohol)	45.	2,2,5-Trimethyl-Hexane
21.	Hexamethyl-Heptane	46.	2,3,4-Trimethyl-Hexane
22.	Hexana1	47.	2,2,6-Trimethyl-Octane
23.	Hexanedioic Acid	48.	2,3,6-Trimethyl-Octane
24.	10-Methyl-Eicosane	49.	Undecane
25.	3-Methyl-5-Propyl-Nonane		

1	 <p>POSSIBLE STRUCTURE</p>	2	 <p>POSSIBLE STRUCTURE</p>	3	$\text{CH}_2(\text{CH}_2)_2\text{CH}_3$ $\text{H}_3\text{C}-(\text{CH}_2)_3-\text{CH}_2-(\text{CH}_2)_3-\text{CH}_3$
4		5		6	$\text{H}_3\text{C}-(\text{CH}_2)_8-\text{CH}_3$
7		8	$\text{H}_3\text{C}-\text{C}(\text{CH}_3)_2-(\text{CH}_2)_7-\text{CH}_3$	9	$\text{H}_3\text{C}-\text{CH}_2-\text{C}(\text{CH}_3)_2-(\text{CH}_2)_2-\text{C}(\text{CH}_3)_2-(\text{CH}_2)_2-\text{CH}_3$

10	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-(\text{CH}_2)_4-\text{CH}_3 \\ \\ \text{CH}_3 \end{array} $	11	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{CH}_2-\text{C}-(\text{CH}_2)_3-\text{CH}_2 \\ \qquad \qquad \\ \text{H} \qquad \qquad \text{CH}_3 \end{array} $ <p>or 3-methyl-octane</p>	12	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{CH}_2-\text{C}-(\text{CH}_2)_3-\text{C}-\text{CH}_2-\text{CH}_3 \\ \qquad \qquad \qquad \qquad \\ \text{H} \qquad \qquad \text{CH}_3 \end{array} $
13	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-(\text{CH}_2)_5-\text{C}-\text{CH}_3 \\ \qquad \qquad \\ \text{H} \qquad \qquad \text{CH}_3 \end{array} $	14	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{CH}_2-\text{C}-(\text{CH}_2)_2-\text{C}-(\text{CH}_2)_4-\text{CH}_3 \\ \qquad \qquad \qquad \qquad \\ \text{H} \qquad \qquad \text{CH}_3 \end{array} $	15	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{CH}_2-\text{C}-(\text{CH}_2)_4-\text{C}-(\text{CH}_2)_2-\text{CH}_3 \\ \qquad \qquad \qquad \qquad \\ \text{H} \qquad \qquad \text{CH}_3 \end{array} $
16		17	$ \text{H}_3\text{C}-(\text{CH}_2)_{18}-\text{CH}_3 $	18	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-(\text{CH}_2)_3-\text{C}-\text{CH}_2-\text{CH}_3 \\ \qquad \qquad \\ \text{H} \qquad \qquad \text{CH}_2\text{CH}_3 \end{array} $

19	$\text{H}_3\text{C}-(\text{CH}_2)_{15}-\text{CH}_3$	20	$\begin{array}{c} \text{H} \\ \\ \text{H}_3\text{C}-(\text{CH}_2)_{14}-\text{C}-\text{OH} \\ \\ \text{H} \end{array}$	21	$\begin{array}{ccccccc} & \text{CH}_3 & & \text{H} & & \text{CH}_3 & & \text{CH}_3 \\ & & & & & & & \\ \text{H}_3\text{C}- & \text{C} & - & \text{C} & - & \text{C} & - & \text{CH}_2-\text{CH}_3 \\ & & & & & & & \\ & \text{CH}_3 & & \text{CH}_3 & & \text{H} & & \text{CH}_3 \end{array}$ one of many possible structures
22	$\begin{array}{c} \text{O} \\ \\ \text{H}_3\text{C}-(\text{CH}_2)_4-\text{C}-\text{H} \end{array}$	23	$\begin{array}{c} \text{O} \qquad \qquad \text{O} \\ \qquad \qquad \\ \text{HO}-\text{C}-(\text{CH}_2)_4-\text{C}-\text{OH} \end{array}$	24	$\begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-(\text{CH}_2)_8-\text{C}-\text{H} \\ \\ \text{H} \end{array}$
25	$\begin{array}{c} \text{CH}_3 \qquad \text{H} \\ \qquad \\ \text{H}_3\text{C}-\text{C}-\text{CH}_2-\text{C}-(\text{CH}_2)_3-\text{CH}_3 \\ \qquad \\ \text{H} \qquad \text{CH}_2\text{CH}_2\text{CH}_3 \end{array}$	26	$\begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-\text{H} \\ \\ \text{H} \end{array}$ $\begin{array}{c} \text{H} \\ \\ \text{H}_3\text{C}-(\text{CH}_2)_{10}-\text{C}-\text{OH} \\ \\ \text{H} \end{array}$	27	$\text{H}_3\text{C}-(\text{CH}_2)_{18}-\text{CH}_3$

28	$\text{H}_3\text{C}-(\text{CH}_2)_7-\text{C}=\text{C}-\begin{array}{c} \\ \text{H} \\ \\ \text{H} \end{array}-(\text{CH}_2)_8-\text{OH}$	29		30	
31	$\begin{array}{c} \text{H} \\ \\ \text{H}_2\text{C}=\text{C}-(\text{CH}_2)_{10}-\text{C}=\text{CH}_2 \\ \\ \text{H} \end{array}$	32	$\begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-(\text{CH}_2)_3-\text{C}-\text{CH}_3 \\ \quad \\ \text{CH}_3 \quad \text{CH}_3 \end{array}$ <p>one of many possible structures</p>	33	$\begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-(\text{CH}_2)_2-\text{C}-\text{CH}_3 \\ \quad \\ \text{CH}_3 \quad \text{CH}_3 \end{array}$
34	Same as 33	35	$\begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-\text{CH}_2-\text{C}-\text{CH}_3 \\ \quad \\ \text{CH}_3 \quad \text{CH}_3 \end{array}$	36	$\begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-\text{CH}_2-\text{C}-\text{H} \\ \quad \\ \text{CH}_3 \quad \text{CH}_3 \end{array}$

37	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-(\text{CH}_2)_4-\text{C}-\text{CH}_3 \\ \quad \\ \text{CH}_3 \quad \text{H} \end{array} $	38	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-(\text{CH}_2)_6-\text{C}-\text{CH}_3 \\ \quad \\ \text{CH}_3 \quad \text{H} \end{array} $	39	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-\text{CH}_2-\text{C}-\text{CH}_3 \\ \quad \quad \\ \text{H} \quad \text{H} \quad \text{CH}_3 \\ \text{CH}_3 \quad \text{CH}_3 \\ \quad \\ \text{C}-(\text{CH}_2)_4-\text{C}-\text{CH}_3 \\ \quad \\ \text{H} \quad \text{H} \end{array} $
40	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-(\text{CH}_2)_3-\text{C}-\text{CH}_2-\text{CH}_3 \\ \quad \quad \\ \text{H} \quad \text{CH}_3 \quad \text{H} \\ \text{CH}_3 \quad \text{H} \\ \quad \\ \text{C}-\text{CH}_2-\text{C}-\text{CH}_3 \\ \quad \\ \text{H} \quad \text{CH}_3 \end{array} $	41	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-\text{CH}_2-\text{C}-(\text{CH}_2)_2-\text{CH}_3 \\ \quad \quad \\ \text{CH}_3 \quad \text{CH}_3 \quad \text{H} \end{array} $	42	$ \begin{array}{c} \text{CH}_3 \quad \text{H} \quad \text{CH}_3 \\ \quad \quad \\ \text{H}_3\text{C}-\text{C}-\text{C}-\text{C}-(\text{CH}_2)_2-\text{CH}_3 \\ \quad \quad \\ \text{H} \quad \text{CH}_3 \quad \text{H} \end{array} $
43	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-(\text{CH}_2)_3-\text{C}-\text{CH}_2-\text{CH}_3 \\ \\ \text{CH}_3 \end{array} $	44	$ \begin{array}{c} \text{CH}_3 \quad \text{CH}_3 \\ \quad \\ \text{H}_3\text{C}-\text{C}-\text{CH}_2-\text{C}-\text{CH}_2-\text{CH}_3 \\ \quad \\ \text{CH}_3 \quad \text{H} \end{array} $	45	$ \begin{array}{c} \text{CH}_3 \quad \text{CH}_3 \\ \quad \\ \text{H}_3\text{C}-\text{C}-(\text{CH}_2)_2-\text{C}-\text{CH}_3 \\ \quad \\ \text{CH}_3 \quad \text{H} \end{array} $

46	$ \begin{array}{c} \text{CH}_3 \quad \text{H} \quad \text{CH}_3 \\ \quad \quad \\ \text{H}_3\text{C}-\text{C}-\text{C}-\text{C}-\text{CH}_2-\text{CH}_3 \\ \quad \quad \\ \text{H} \quad \text{CH}_3 \quad \text{H} \end{array} $	47	$ \begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{C}-\text{C}-\text{CH}_2-\text{CH}_3 \\ \quad \\ \text{CH}_3 \quad \text{H} \end{array} $	48	$ \begin{array}{c} \text{CH}_3 \quad \text{H} \quad \text{CH}_3 \\ \quad \quad \\ \text{H}_3\text{C}-\text{C}-\text{C}-\text{C}-\text{CH}_2-\text{CH}_3 \\ \quad \quad \\ \text{H} \quad \text{CH}_3 \quad \text{H} \end{array} $
49	$\text{H}_3\text{C}-(\text{CH}_2)_9-\text{CH}_3$	50		51	
52		53		54	

Appendix C

Glossary Of Chemical Nomenclature

GLOSSARY OF CHEMICAL NOMENCLATURE

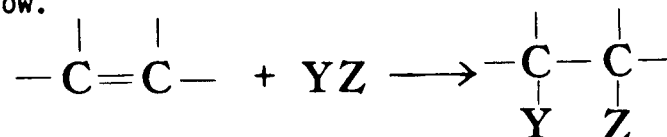
Activation

Energy

Activation energy is defined as the minimum amount of energy required for a chemical reaction to occur; the source of which is the kinetic energy associated with the molecules.

Addition

An addition reaction occurs when two molecules combine to form a single-molecular product. Addition reactions occur in compounds that contain atoms with multiple bonds (such as a carbon-carbon double bond) where the weaker of the two bonds is broken when a reagent combines with the organic molecule as shown below.

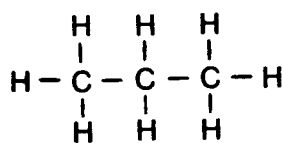


Alcohols

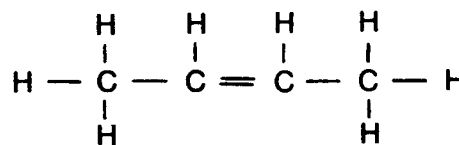
Alcohols are compounds of the general formula, ROH, where R is any alkyl group; this group may be open-chain or cyclic, and may contain a double bond, a halogen atom, or an aromatic ring. Alcohols are polar molecules since they contain a hydroxyl (OH) group.

Aliphatics

Aliphatic compounds include open-chain structures and cyclic compounds resembling the open-chain structures as shown below. Aliphatics react via addition at multiple bonds and by free-radical substitution at other points along their chain.



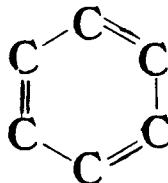
Propane



2-Butene

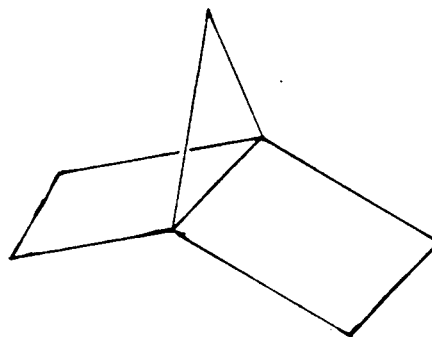
Aromatics

Aromatic compounds are benzene and compounds that resemble benzene in structure and chemical behavior. The structure of benzene is shown below.



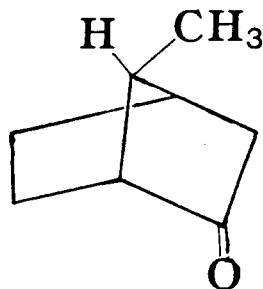
Bicyclic

Bicyclic compounds contain two rings which share two carbon atoms as indicated below for bicycloheptane.



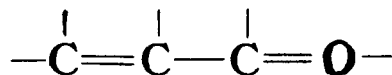
Bridged Structure

Bridged structures are rings fused through nonadjacent atoms. An example of the bridged compound, endo-7-methyl-2-norcamphor, is shown below.



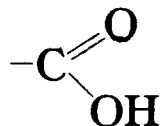
Carbonyls

A carbonyl structure contains a carbon-carbon double bond and a carbon-oxygen double bond separated by just one carbon-carbon single bond as shown below. The carbon-carbon double bond serves as a source of electrons for electrophilic reactions.



Carboxylic Acid

Carboxylic acids are a family of organic compounds that contain the carboxyl group (shown below) attached to either an aliphatic (alkyl group, RCOOH) or an aromatic (aryl group, ArCOOH) group. Carboxylic acids are polar and can form hydrogen bonds with each other and with other kinds of molecules.



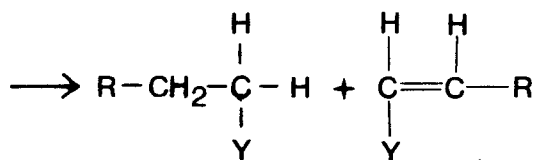
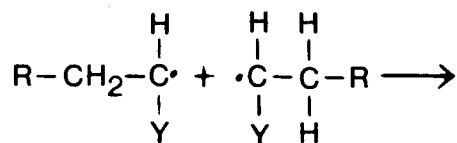
Cyclic

Aliphatics

Cyclic aliphatics are hydrocarbons whose carbon atoms are arranged to form rings. They are generally prepared in two stages: conversion of open-chain compounds into compounds that contain rings, and conversion of the cyclic compound into a desired type.

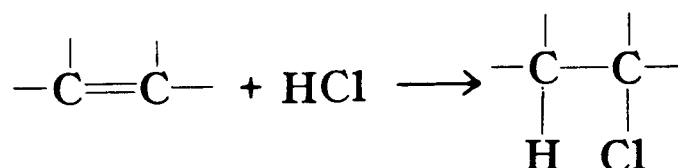
Disproportionation

A disproportionation reaction is a termination step in a free radical chain reaction. In a disproportionation reaction radicals are consumed without forming additional free radicals to promote the continuing reaction. A simple disproportionation reaction involving the transfer of hydrogen is shown below. Note that free radicals are consumed.



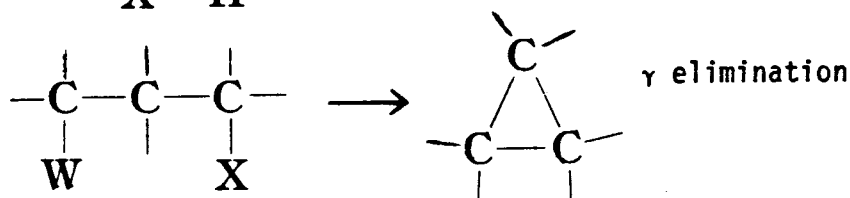
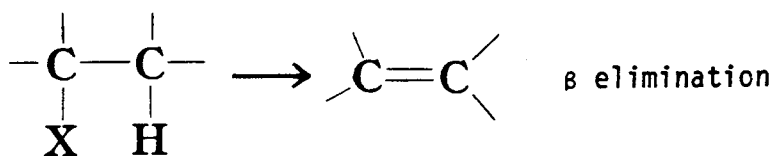
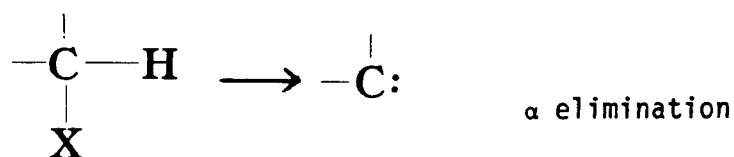
Electrophilic Addition

Electrophilic addition is a reaction involving a carbon-carbon double bond in which an acidic reagent is added to form a single-molecular product. Reagents that are electron-deficient, or acidic, seek the pair of electrons that are held less tightly by the carbon-carbon double bond. A simple electrophilic addition reaction involving hydrochloric acid (HCl) is shown below.



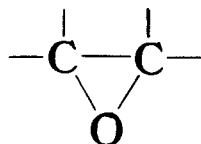
Elimination

There are several types of elimination reactions. In an α elimination both groups are lost from the same atom to give a carbene (or nitrene), a reactive intermediate. When two groups are lost from adjacent atoms so that a new double (or triple) bond is formed, the reaction is called β elimination. In a γ elimination, a three-membered ring is formed. Examples of α , β , and γ eliminations are shown below.



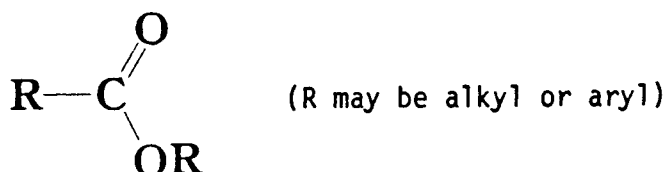
Epoxide

An epoxide is a compound that contains a three-member ring which includes two carbon and one oxygen atoms as shown below. The epoxides are ethers, however, the three-membered ring gives them unusual properties.



Esters

Esters are compounds closely related to carboxylic acids except that the hydrogen of the carboxylic group is replaced by an alkyl or aryl group as shown below. Esters undergo nucleophilic substitution typical of carboxylic acid derivatives.

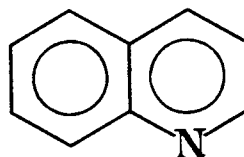


Fragmentation

Fragmentation is a reaction in which carbon is the positive leaving group in an elimination.

Heterocyclics

Heterocyclics are compounds that contain a ring made up of more than one kind of atom as indicated below for quinoline. Some common five-member rings include pyrrole, furan, and thiophene, which undergo electrophillic substitution rather than other types of reactions. Other heterocyclic structures are six-member rings, such as pyridine, and fused rings.



Homolysis

Homolysis is bond-breaking in which one of the two bonding electrons goes to each fragment from the breaking of a covalent bond.

Hydrocarbons

Hydrocarbons are organic compounds containing only hydrogen and carbon. They are divided into two structural categories--aliphatic, which are open chain compounds, and aromatic, which have a ring structure and resemble benzene in chemical behavior.

Iso-electronic

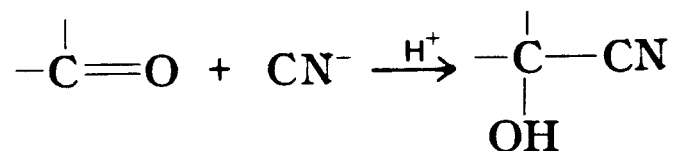
Isoelectronic molecules are molecules containing the same number of valence electrons.

Isomers

Isomers that differ from one another only in the way the atoms are oriented in space, but are not mirror images (enantiomers) of each other, are called diastereomers. Those which owe their existence to hindered rotation about double bonds are called geometric isomers.

Nucleophilic Addition

Nucleophilic addition is a reaction involving a carbon-oxygen or carbon-carbon double bond in which a base is added to form a single-molecular product. Electron-rich nucleophilic reagents, or bases, are attracted to the electron-deficient carbonyl carbon prompting the formation of a bond. When a carbon-oxygen bond is involved, the tendency of oxygen to acquire electrons causes the reactivity of the C=O group toward bases. A simple nucleophilic addition reaction is shown below.



Olefin

Olefins are a family of unsaturated, chemically active hydrocarbons with one carbon-carbon double bond. Olefins include ethylene and propylene.

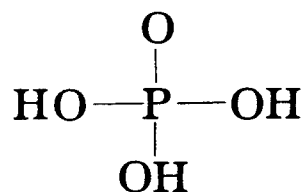
Oxidation

In inorganic chemistry, oxidation involves the loss of electrons and a corresponding increase in the oxidation number of a compound. In combustion, oxidation occurs when an organic compound such as methane is converted to carbon dioxide and water. In this case, the carbon of methane is oxidized (gains oxygen) to carbon dioxide. An oxidative environment is defined as being rich in oxygen atoms and hydrogen-deficient.

Phosphate

Ester

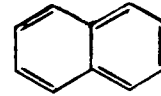
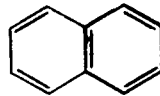
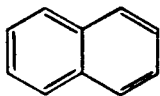
Phosphoric acid (shown below) contains three hydroxy groups and forms esters in which one, two, or three of these hydroxy groups (OH) are replaced by alkoxy groups (OR).



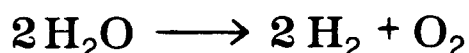
Polynuclear

Aromatics

Polynuclear aromatics are compounds that have two or more aromatic rings that share a pair of carbon atoms. Rings that are part of this structure are considered fused. The simplest and most important fused-ring aromatic, naphthalene, C_{10}H_8 , is a hybrid of the three structures shown below.



<u>Pyrolysis</u>	In pyrolysis the decomposition of a compound occurs by action of heat with no other reagent present. In combustion, starved air conditions are referred to as pyrolysis conditions.
<u>Radical</u>	A radical is an atom or anion possessing an unpaired electron in its valence shell that makes it reactive.
<u>Reducing Conditions</u>	An environment that is rich in hydrogen atoms and oxygen-deficient is reducing. When an organic compound is reduced, it gains hydrogen and/or loses oxygen.
<u>Strained</u>	A strained condition occurs when a molecule's bonds are forced to make abnormal angles, resulting in a higher energy level than would be the case in the absence of angle distortions. Such strained structures are more unstable than structures with normal bond angles.
<u>Thioether</u>	A thioether is a derivative of thiol which contains the sulfhydryl group (-SH). Thiols are sulfur analogs of alcohols and are easily oxidized.
<u>Unimolecular</u>	A unimolecular process involves only one reactant molecular. An example is the electrolysis of water as shown below.



Sources: March, J. 1985. Advanced Organic Chemistry 3rd ed., John Wiley and Sons: New York, N.Y. 1985. Morrison, R.T. and R.N. Boyd. 1973. Organic Chemistry 3rd ed. Allyn and Bacon: Boston, Massachusetts. 2114D

10/07/87

Appendix D

Response To Shell Oil And EPA
Region VIII Comments For The Report Titled
"Bench-Scale Laboratory Incineration Of
Section 36 Wastes" (Expansion Program)

Response To Shell Oil Comments

RESPONSES TO SHELL OIL COMPANY COMMENTS

A copy of the original letter of the Shell Oil comments is on file at PMO.

Only comments applicable to the report titled "Bench-Scale Laboratory Incineration of Section 36 Wastes (Expansion Program) at Rocky Mountain Arsenal" dated March 1988 are presented here. Comments have been incorporated into the final white cover version of the subject report dated September 1988.

COMMENT NO. 1

Page 1-1, first paragraph of 1.1. It is incorrect to state that "...gathering information on the technical and economic aspects of incineration/thermal treatment...(is) the initial step towards developing a broad remedial action alternative for RMA."

RESPONSE TO COMMENT NO. 1

The purpose of Task 17 is to evaluate incineration/thermal treatment for use as a means of remediation for Basin F and Section 36 wastes. To do this, technical information must be collected and the alternatives evaluated from an economic standpoint. Final evaluation of incineration will take place during the Arsenal-wide feasibility study.

COMMENT NO. 2

Page 2-1, first paragraph under 2.2.1. In the second sentence, liquid wastes should be aqueous wastes. In the last sentence, only during the initial period of Basin F use did the waste first pass through Basin A.

RESPONSE TO COMMENT NO. 2

The above changes have been incorporated into the final white cover version of the subject report.

COMMENT NO. 3

Table 2.2-1 and 2.2-2. The analytical results in these two tables, for Source 36-1 and 36-3, respectively, are identical.

RESPONSE TO COMMENT NO. 3

The content of the tables has been corrected for the final white cover version of the subject report.

COMMENT NO. 4

Page 2-13, Field Sampling Program. The native soil type for each of the samples tested should be characterized for future reference. Soil type may be an important variable requiring future study.

RESPONSE TO COMMENT NO. 4

The importance of soil type is well established with regard to such factors as particle size, moisture, angle of repose, ash fusion characteristics, and the elemental composition including trace metals. However, such characterization is not a part of Task 17. Such work would fit well into a pilot plant program.

COMMENT NO. 5

Page 2-16, Table 2.4-1. The levels of organic contamination in sample 36-1-3204A, which was used in the laboratory program for organic experiments, are exceedingly high and are representative of only a small fraction of contaminated soil in the basins and elsewhere on the RMA.

RESPONSE TO COMMENT NO. 5

The samples with the highest concentrations were used in order to have a sufficient concentration of POHCs to determine if a 99.99+ percent DRE is being achieved. The other samples had insufficient concentrations of POHCs.

COMMENT NO. 6

Page 3-6, first paragraph. What is meant by the parenthetical the selected technology in the fifth sentence? In the table, the units of maximum Linear Heating Rate should be °C per minute.

RESPONSE TO COMMENT NO. 6

The parenthetical material refers to the rotary kiln that was selected for further evaluation as part of Task 17. However, the statement could also apply to the fluidized bed or other technologies. The text has been changed in the final white cover version of the subject report to reflect this point.

The units in the table have been changed to °C/minute in the final white cover version of the subject report.

COMMENT NO. 7

Page 4.2, last paragraph. The assumption that insignificant loss of organics occurs at 105°C over 60 minutes may well be invalid, especially if soil moisture content is high, as is claimed. Shell's studies indicate significant loss of dieldrin at low temperatures (110 - 150°C) when soil moisture is high. What was the purpose of maintaining the sample at 105°C for 60 minutes? Were any tests done to see if loss of organics under these conditions was indeed insignificant?

RESPONSE TO COMMENT NO. 7

The sample was maintained at 105°C in order to prevent the explosive flashing of water to steam in the muffle furnace. It is realized that soil moisture stripping may occur, although this was not evaluated. However, if this did occur, it occurred equally for all samples, thus maintaining the same relative baseline concentrations of organics before actual heatup began.

COMMENT NO. 8

Page 4-9, first paragraph. Why was insufficient material trapped with Basin A material when sufficient material was trapped with Basin F material? Basin A material started with much higher concentrations of contaminants than did Basin F material.

RESPONSE TO COMMENT NO. 8

The Section 36 samples were highly variable in the concentration of organics and not all of the samples had sufficient concentrations of organics. The choice to use Basin F samples was partly due to improper Basin A sample selection and experimental error.

COMMENT NO. 9

Page 4-12, table. What was the primary chamber residence time? Was it varied?

RESPONSE TO COMMENT NO. 9

The primary chamber residence time was 60 minutes at temperature. The residence time was not varied.

COMMENT NO. 10

Page 4-12, 4.5.2 Test Procedure. It would be of use to analyze residue from the primary chamber and residue from the fly ash separator separately to help in determining metals partitioning.

RESPONSE TO COMMENT NO. 10

Separate GC/MS analysis of the residue from the primary chamber and the fly ash separator was not conducted. It is doubtful that the particle size distribution and temperature in the fly ash separator would have been sufficient to detect metal partitioning beyond mercury and arsenic.

COMMENT NO. 11

Page 5-6, Figure 5.1-1. Is this curve for pure aldrin? If so, it does not reflect the effect of soil moisture "stripping" at lower temperatures.

RESPONSE TO COMMENT NO. 11

The curve is for pure aldrin. As indicated in the comment, it does not reflect soil moisture stripping.

COMMENT NO. 12

Page 5-7, table. Sample 3204 (which showed high weight loss at low temperature) also had very high concentrations of organics, which would account for the higher weight loss.

RESPONSE TO COMMENT NO. 12

This is recognized and the table has been changed in the final white cover version of the subject report to reflect that sample 3204 contained between 6 and 19 percent organics.

COMMENT NO. 13

Page 5-8, Section 5.3. These results are in direct contradiction of results from organic volatilization experiments. The organic volatilization experiments say that a minimum of 500°C is needed to volatilize organics, while ramping experiments show that 300°C is sufficient. Can this discrepancy be explained?

Data for CPMS indicate most volatilization takes place between 200 - 300°C, not 100 - 200°C as stated.

Data for aldrin show significant volatilization between 100 - 200°C as well as 200 - 300°C. The effect at low temperature could be explained by soil moisture "stripping". Perhaps CPMS is held more tightly to soil (because of sulfur?) so soil moisture "stripping" has less impact.

RESPONSE TO COMMENT NO. 13

The ramping experiment was to show the relative rate of organic volatilization. Because of problems outlined in the text, it was not possible to calculate a DRE or to complete a mass balance, and as such not possible to select an optimum temperature. The text of the final white cover version of the subject report has been corrected to reflect the inconsistencies indicated.

COMMENT NO. 14

Page 5-8, Section 5.4. The temperature profile is only across half of the primary chamber, so no conclusions can be drawn about temperature profile across the entire primary chamber. Temperature could continue to increase across the chamber, or it could remain the same or decrease.

RESPONSE TO COMMENT NO. 14

With the thermocouples available, it was not possible to profile the entire primary chamber during operation. However, the area profiled represents the location of the cool regions.

COMMENT NO. 15

Page 5-11. It would have been useful to see bench-scale tests below 450°C, since DRE at 450°C was still greater than 99.99%.

RESPONSE TO COMMENT NO. 15

It is agreed that it would have been useful to conduct experiments below 450°C in the primary chamber.

COMMENT NO. 16

Page 5-25, first paragraph. Qualitatively, one could conclude that significant volatilization occurred only for arsenic, mercury, and sodium.

RESPONSE TO COMMENT NO. 16

It is agreed that this is the only qualitative conclusion possible from the data.

COMMENT NO. 17

Page 5-27, first line. Why weren't samples from bench-scale tests split for analysis of both TCLP and EP Tox?

It would also have been useful to run EP Tox on residues from low temperature treatment.

RESPONSE TO COMMENT NO. 17

Samples from the bench-scale apparatus or split samples could not be used since all the material was required for extraction for the GC/MS analysis for POHCs. Bench-scale runs were not conducted for the purpose of supplying material for the EP Toxicity or TCLP analysis. It is agreed that analysis on low temperature material would have been useful.

COMMENT NO. 18

Page 6-2, point (4. Since high temperature (900°C) generated most PICs (phenol), and target organics could be removed sufficiently at lower temperature (500°C), wouldn't the logical conclusion be that 500°C is the optimum temperature for the primary temperature.

Low temperature desorption of organics from solids offers many advantages over high temperature incineration. Shell's studies on low temperature desorption continue to show promise for treatment of certain contaminated soils on the RMA. In general the results of this Task 17 investigation support further investigations of low temperature desorption.

RESPONSE TO COMMENT NO. 18

It is agreed that a 500°C or even 600°C soil temperature may be sufficient to volatilize the organics for destruction in an afterburner. However, in a rotary kiln the gas and solids approach temperatures are on the order of 150 to 250°C, which still indicates a gas temperature on the order of 800 to 900°C. It is agreed that low temperature volatilization deserves further investigations.

Response To EPA Region VIII Comments

RESPONSES TO THE UNITED STATES ENVIRONMENTAL PROTECTION AGENCY,
REGION VIII COMMENTS

A copy of the original letter of the EPA Region VIII comments is on file at PMO.

Only comments applicable to the report titled "Bench-Scale Laboratory Incineration of Section 36 Wastes (Expansion Program) at Rocky Mountain Arsenal" dated March 1988 are presented here.

General Comments

COMMENT NO. 1

A baseline calibration of the device using processing materials with known combustion characteristics would aid in the interpretation of data collected from test burns. This kind of calibration also models PIC generation and identifies combustion failure modes such as inefficient heat transfer to solids and chilling or inadequate mixing of the gas phase.

RESPONSE TO COMMENT NO. 1

Baseline runs using Basin F soil were conducted at the beginning of the program to see the level of recovery that could be obtained. Materials with known combustion characteristics were not modeled.

COMMENT NO. 2

The data collected from the tests for the volatilization of metals will only be useful for indicating the upper limits for operation, below which metal loss from the solid phase into the gas phase is minimized. Overall, the tests performed for the metals could have been enhanced by

including more definitive studies of the feed material and burn environment. This is because transfer from the burning mass into the gas stream is sensitive to:

- 1) The chemical and physical form in which the element is found in the waste.
- 2) The chemical (particularly halogen and oxygen concentrations) and physical characteristics of the incineration environment.

Items (1) and (2) were not treated in depth as variables in the metals tests making use of the resulting data limited.

RESPONSE TO COMMENT NO. 2

Metal partitioning studies were attempted. However, wide variations in the concentrations of metals produced results of little use. Metal partitioning experiments were not attempted in the bench-scale unit because all the sample was used for the GC/MS analysis of the residue. The characterization of the physical and chemical forms of the metals and halogens in the waste was not a part of the investigation. Metal partitioning is an important parameter and would be a part of any future pilot plant program.

COMMENT NO. 3

Vaporization of metals can cause either (a) gaseous emissions or (b) condensation of toxic elements onto particulate nuclei, both processes of which can create small particles in the gas stream. These small particles are not easily collected by pollution control equipment. Therefore, tests for particulate size distributions of elements carried in the gas stream should have been performed during the volatilization tests.

RESPONSE TO COMMENT NO. 3

Particulate size distributions were not conducted. It is felt that the quantity and characteristics of the particulate were not representative of a full scale or pilot system. Particulate size distribution would be a part of any future pilot plant program.

COMMENT NO. 4

No attempt was made to measure and then apply the vaporization potential of toxic elements in the metal-bearing feeds to prediction of enrichment of volatile metals as particulates that could subsequently be emitted from the incinerator. Studies by Gerstle and Albrinck (1982) suggest that enrichment is negligible below 815°C but significant above 925°C. Burn temperatures above 900°C may have to be re-evaluated based on their findings.

RESPONSE TO COMMENT NO. 4

It is agreed that enrichment is an important concern that has been well documented for both coal and municipal solid waste combustion. However, such studies are beyond the capabilities of the equipment used as well as the scope of the subject research. Enrichment could be more accurately evaluated during a future pilot plant program.

Specific Comments

COMMENT NO. 1

Page 3-6, first paragraph. Highly turbulent flow is characteristic of the gas stream in the quartz tubes of the afterburner. The turbulence produced means that spatial variation in velocities will occur with the effect that some emissions will experience residence times much lower than the mean residence times used to make assumptions about the effectiveness of burn time. The low-end tail of residence times should be characterized to ensure complete burnout of toxic compounds.

RESPONSE TO COMMENT NO. 1

The variations in velocity and the determination of the statistical distribution in the quartz tube of the afterburner were not evaluated. Since most DREs were well above 99.99 percent and no POHCs were found as PICs, our data should be viewed as a worst case with regard to incineration conditions.

COMMENT NO. 2

Page 3-6, Muffle Furnace System. Actual heatup rates for the muffle furnace were evaluated because of their importance to organic volatilization. However, the heatup rate will decrease as feed material moisture increases, with a subsequent effect on the effectiveness of residence times. Therefore, the effect of soil moisture content must be modeled along with heatup rates so that appropriate residence times according to moisture content can be defined.

RESPONSE TO COMMENT NO. 2

Soil moisture content is only important up to the phase change of liquid to gaseous water due to the higher heat capacity of water and its increased thermal conductivity. However, above 100°C this advantage is lost. Also any potential advantage in reducing the heatup rate, due to higher moisture content, has to be viewed in light of increased gas volumes in the incinerator, reduced gas residence times, reduced flame temperatures, and reduced radiative heat transfer.

COMMENT NO. 3

Page 5-25, first and second paragraphs. The discussion of volatilization of arsenic, mercury, sodium, copper, etc., refers to metals in their elemental states. It is improbable that these metals do exist in their elemental states. They will instead be bound in clays, carbonates, or as salts. Therefore any evaluation of results

and prediction of behavior of metals being released into the vapor state must instead take into account the manner in which the release of metals from these bound states occurs. This indicates that the feed material should be studied to determine how the metals are bound in the contaminated soils.

RESPONSE TO COMMENT NO. 3

The importance of characterizing the feed material is recognized but outside the scope of this research.



Appendix E

References

APPENDIX E
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